Bereskin & Parr
INTELLECTUAL PROPERTY LAW

Appl. No	:	09/916,247	Confirmation No.: 9131
Applicant	:	COTE et al.	
Filed	:	July 30, 2001	
Title	:	CHEMICAL CLEANING BACKWASH FOR NORMALLY IMMERSED MEMBRANES	
TC./A.U.	:	1723	
Examiner	:	MENON, Krishnan S.	
Docket No.	:	4320-347	
Customer No.	:	001059	

Board of Patent Appeals and Interferences
United States Patent and Trademark Office
P. O. Box 1450
Alexandria, Virginia 22313–1450

July 25, 2007

BRIEF IN SUPPORT OF APPEAL

Real Party in Interest

The Real Party in Interest in the present Appeal is Zenon Technology Partnership, the assignee. Zenon Technology Partnership is a partnership between GE Betz Canada Company and 1244734 Alberta ULC. GE Betz Canada Company is related to GE Betzdearborn Canada Company, GE Betz Inc., MRA Investments Inc., MRA Systems Inc., GE Investments Inc. and General Electric Company. Other companies may have a non-controlling interest in one or more of these companies.

Related Appeals and Interferences

This application was remanded to the Examiner after a decision in this application in Appeal 2006-2492, decided on February 16, 2007.

Application Serial No. 09/425,234 is the parent of this application. Application Serial Nos. 10/377,647 and 10/461,687 are continuing applications in the family of Application Serial No. 09/425,234. The real party in interest, Zenon Technology Partnership, owns all of these applications.

An appeal in Application Serial No. 09/425,234, Appeal 2007-0362, was decided on March 23, 2007. That application has since been abandoned.

An appeal in Application Serial No. 10/377,647 is pending.

An appeal in Application Serial No. 10/461,687, Appeal 2006-2898, was decided on February 28, 2007. That decision remanded Application Serial No. 10/461,687 to the Examiner, who then rejected all claims. A further Notice of Appeal has been filed.

The appeals or decisions described above may directly affect or have a bearing on the Board's decision in this appeal.

There are no related interferences.

Status of the Claims

Claims 1-25 have been cancelled. Claims 26-36 are pending and the subject of this appeal.

Status of Amendments

No amendments have been filed after the Office Action mailed February 26, 2007. The claims are the same now as they were during the previous appeal in this application, Appeal 2006-2492, decided on February 16, 2007.

Summary of the Claimed Subject Matter

The Applicant's invention, as defined by claim 26, relates to a process for filtering water containing solids with membranes in a tank (page 5, line 19 to page 6, line 9; "liquid feed 14" or "tank water 22", "membranes 24" and "tank 20" shown in Figure 1).

The basic process involves five steps, (a) filling the tank with feed water to immerse the membranes (page 6, lines 1-2); (b) generating a filtered permeate at a permeate outlet and a retentate in the tank (page 7, line 9 to page 8, line 12; "permeate 36", "permeate outlet 38", "retentate 46" shown in Figure 1); (c) aerating the membranes to dislodge

solids from the membranes (page 8, lines 13-22); (d) backwashing the membranes (page 8, line 23 to page 9, line 6); and, (e) draining the tank of the retentate (page 7, lines 26-29; page 8, lines 2-6).

These steps, (a) through (e), are performed in a repeated cycle. However, an additional step (f) of wetting the membranes with a cleaning chemical is performed in some or all of the cycles. Step (f) is performed at least once a week after or while draining the tank in a first cycle and without returning to permeation before starting a subsequent cycle (page 9, lines 19-24; page 18, line 21 to page 19 line 3). Because step (f) occurs within a cycle and at least once a week, a cycle, that is steps (a) through (e), also occurs at least once a week. Accordingly, a step of draining the tank of retentate (step (e)) occurs at least once a week.

Claim 28 modifies claim 26 in two ways. Firstly, claim 28 introduces a new step of performing recovery cleanings to increase the permeability of the membranes. Secondly, claim 28 states that the steps of claim 26 are performed between the recovery cleanings to reduce the rate of a decline in permeability of the membranes between the recovery cleanings. These aspects of the invention are discussed, for example, at page 17, line 21 to page 18, line 5. As discussed therein, the concentration and duration of the chemical wetting (step (f)) of claim 26 may be chosen such that the permeability of the membranes continues to decline over an extended period of time even in the presence of the chemical wetting step, but the rate of this decline is reduced. A recovery cleaning is performed at the end of such a period of time to restore the permeability of the membranes. Accordingly, the cleanings of step (f), integrated into a filtration cycle as described in claim 26, are insufficient on their own to preserve permeability of the membranes over their service life (which is hopefully many years), but reduce the frequency at which recovery cleanings would otherwise be required (page 9, lines 7-17).

The other claims further define the invention of claims 26 or 28. Claim 27 shortens the frequency of the filtration cycles, including steps of draining retentate and wetting the membranes with a cleaning chemical, to one day or less (page 18, line 27 to page 19, line 3). Claims 29 and 30 describe the concentration of the cleaning chemical and duration of the wetting of step (b) of claim 26 (page 16, line 28 to page 17, line 20; page 18 lines 6-20). Claim 31 describes the frequency of the recovery cleanings of claim 28 (page 17, line 27). Claim 32 indicates that the process is used for producing drinking water and defines the class of cleaning chemical (page 9. lines 26-28; page 18, lines 6-9). Claim 33 states that the cleanings of step (f) of claim 26 are performed regularly and at about the same effectiveness (page 18, lines 19-20). Claim 34 states that the membranes are backwashed with permeate after being wetted with the cleaning chemical but before starting a new cycle, the effect of which is to rinse the permeate side of the membranes before withdrawing more permeate in the next cycle (page 10, lines 9-11). Claim 35 defines a method of delivering chemicals to the membranes, namely mixing a cleaning chemical into water flowing to the permeate side of the membranes (page 16, lines 12-16). Claim 36 describes hollow fiber membranes (page 6, line 24 to page 7, line 4; "membranes 24" as shown in Figures 1-4).

Grounds of Rejection to be Reviewed on Appeal

I. Statutory Double Patenting

Claims 26-29, 31 and 33 were provisionally rejected for statutory double patenting in relation to claims 1 to 6 of co-pending Application No. 11/106,681.

II. Obviousness-type Double Patenting

Claims 26-36 were provisionally rejected for obviousness-type double patenting in relation to claims 7-29 of Application No. 11/106,681.

III. The Section 103(a) Rejection of Claims 26-36 over Smith in view of Del Vecchio

Claims 26-36 (all pending claims) were rejected as being obvious over Smith et al. US Patent No. 5,403,479 (hereinafter "Smith") in view of Del Vecchio et al. US Patent No. 6,331,251 (hereinafter "Del Vecchio").

IV. The Section 103(a) Rejection of Claims 26-28, 31 and 34-36 over Del Vecchio

Claims 26-28, 31 and 34-36 were rejected as being obvious over Del Vecchio applied alone.

V. The Section 103(a) Rejection of Claims 29, 30, 32 and 33 over Del Vecchio in view of Smith

Claims 29, 30, 32 and 33 (all pending claims not rejected under Del Vecchio alone) were rejected as being obvious over Del Vecchio as applied to claim 26 in the rejection over Del Vecchio alone, but further in view of Smith.

ARGUMENT

I. Statutory Double Patenting

Claims 26-29, 31 and 33 were provisionally rejected for statutory double patenting in relation to claims 1 to 6 of co-pending Application No. 11/106,681. Claims 1 to 6 were cancelled from Application No 11/106,681 by preliminary amendment on May 14, 2007 without being replaced by any new or amended claim. The Appellant submits that this rejection no longer applies.

In the event that this grounds of rejection remains, the Appellants submit that this is a provisional rejections in relation to an application (11/106,681) filed after the application under appeal. If this or other provisional rejections are the only

rejections sustained on this appeal, the Appellant submits pursuant to MPEP 804, Parts IB 1 and 2, that the provisional rejections should be withdrawn in this application and the provisional rejections in application 11/106,681 converted into non-provisional rejections, or that this application be remanded to the Examiner to do so.

II. Obviousness-type Double Patenting

Claims 26-36 were provisionally rejected for obviousness-type double patenting in relation to claims 7-29 of Application No. 11/106,681. A terminal disclaimer to obviate this rejection was filed with the Notice of Appeal in this application.

In the event that this grounds of rejection remains, the Appellants submit that this is a provisional rejections in relation to an application (11/106,681) filed after the application under appeal. If this or other provisional rejections are the only rejections sustained on this appeal, the Appellant submits pursuant to MPEP 804, Parts IB 1 and 2, that the provisional rejections should be withdrawn in this application and the provisional rejections in application 11/106,681 converted into non-provisional rejections, or that this application be remanded to the Examiner to do so.

III. The Section 103(a) Rejection of Claims 26-36 over Smith in view of Del Vecchio

Claims 26-36 (all pending claims) were rejected as being obvious over Smith et al. US Patent No. 5,403,479 (hereinafter "Smith") in view of Del Vecchio et al. US Patent No. 6,331,251 (hereinafter "Del Vecchio").

Claim 26

The Appellants submit, in brief, that the Examiner's rejection is improper because of the following:

(a) first, the Examiner improperly characterized Smith as merely stating that a step of draining the tank can be eliminated whereas Smith actually described a step of draining the tank as being part of a highly undesirable process and not part of the inventive process described in Smith;

(b) second, the Examiner improperly applied various cases on anticipation to ignore Smith's teaching against a step of draining the tank which is contrary to the alleged obvious combination of Smith and Del Vecchio; and,

(c) third, the Examiner did not establish that element (f) of claim 16 was disclosed in, or made obvious by, either of the cited references.

These arguments will be discussed in greater detail below.

Regarding point (a), Smith '479 does not disclose a process having all of the elements of claim 26. This was acknowledged in the decision of February 16, 2007 in the prior appeal 2006-2492 in this application. The Examiner asserts, however, that "..Smith discusses about draining the tank in detail during cleaning in the "back-ground of the invention", but teaches that draining the tank can be eliminated during the cleaning process ...(col 10 lines 64-68, col 11 lines 22-61)" (see the bottom of page 4 of the Office Action). The Appellant submits that the Examiner's statement mischaracterizes Smith for the reasons discussed below.

The Examiner cited column 10, lines 64-68, which are part of the "Background of the Invention" part of Smith. The entire paragraph included column 10 lines 64-68 is repeated below:

> An obvious drawback of cleaning from the outside of a tube or fiber, rather than from the inside, is that to do so requires a shell. If there is no shell, as in a frameless array such as one disclosed in the '524 array must be removed from the process reservoir in which it operates and immersed in a cleaning solution in another tank. An alternative is to drain the process reservoir and to substitute cleaning solution; then drain the cleaning solution after cleaning, and refill the reservoir. As is evident, this is a highly undesirable alternative.

In the paragraph above, draining the tank is described as an alternative to another available prior art process, which is to remove the membranes and immerse them for cleaning in another tank. The alternative process involving draining the tank is "highly undesirable".

The Examiner also cites column 11, lines 22-61. This passage is part of the "Summary of Invention" portion of Smith. The only reference to draining a tank in that entire passage is in the following sentence at lines 22-29:

> Highly effective cleaning of a module containing an UF or MF membrane having a fouled surface is obtained during an unexpectedly short period, without draining feed (substrate) from the module, by introducing a chosen cleaning fluid into the permeate and recycling it through the lumens at low pressure in the range from about atmospheric but no more than the bubble point of the fiber.

Thus the only reference to draining a tank in the Summary of Invention in Smith says that draining the tank is not part of the invention. Coupled with the statement mentioned above that draining the tank is part of a highly undesirable prior art process, the Appellants submit that Smith teaches away from any use of a tank draining step.

Regarding point (b), the Examiner cites four cases on page 5 of the Office Action for the proposition that a non-preferred or disparaged embodiment is still part of the disclosure of a cited reference. For example, the Examiner states that ".. a reference is no less anticipatory if, after disclosing the invention, the reference then disparages it" and that "The question whether a reference "teaches away" from the invention is inapplicable to an anticipation analysis." While those propositions may be relevant to an anticipation reference where a non-preferred embodiment has all elements of a claim, the decision of February 16, 2007 in Appeal 2006-2492 has already determined that Smith and Del Vecchio do not have all of the elements of any claim pending in this application. Since this is an obviousness rejection, not an anticipation reference, the Appellants submit that the Examiner has improperly directed himself to ignore the teaching in Smith that is contrary to a step of draining a tank.

The Appellants further submit that the teaching in Smith is contrary to any combination with Del Vecchio in particular. All of the steps in the prior art process disparaged in Smith ("to drain the process reservoir and to substitute cleaning solution; then drain the cleaning solution after cleaning, and refill the reservoir" – see column 10, lines 64-68) are actually part of the basic "deep cleaning" process that the Examiner relies on in Del Vecchio. That process, described at column 11, line 47 to column 12, line 48, includes steps of draining substrate (water to be filtered) from a compartment containing the membranes (column 11, lines 52-55), refilling the compartment with a cleaning chemical (column 11, lines 64-65), soaking the membranes for a period of time in the cleaning chemical (column 12, lines 11-16), draining the compartment of cleaning chemical (column 12, lines 39-42), then re-filling the compartment with substrate (column 12, lines 44-48). The Appellants submit that the disparaging comments in Smith thus apply to Del Vecchio. Since Smith seeks to replace such a process, there cannot be any obvious combination of these references. The

Examiner notes that Del Vecchio states (at column 12, lines 49-65) that the last two steps (draining the tank of cleaning chemical and re-filling with substrate) can be avoided in some circumstances, but there is still no reason to combine the process of Del Vecchio with Smith because Smith seeks to avoid all tank draining steps and further teaches against any process in which membranes are cleaned form the outside as in Del Vecchio. In particular, column 11, lines 1 to 19 of Smith describe further disadvantages of cleaning from the outside of the membranes.

Regarding point (c), part (f) of claim 16 states as follows:

> f) wetting the membranes at least once per week with a cleaning chemical having a selected concentration for a selected duration after performing step (b) [permeating] in a first cycle and after or while performing step (e) [draining the tank] in the first cycle, without returning to step (b) in the first cycle and before starting a subsequent cycle.

Part (f) of claim 16 requires wetting the membranes at least once per week. The Examiner argues that Figure 4 of Smith discloses wetting the membranes with a cleaning chemical at least once per week. However, in Figure 4, data points (2), (3) and (4) relate to simple backwashes with water only and there is about a 9 day gap between data points (1) and (5). Accordingly, this argument is incorrect.

Further, part (f) of claim 16 requires wetting the membranes with a cleaning chemical after or while draining the tank and before starting a new cycle. A new cycle will require another step of draining the tank. The tank is therefore drained at least once per week. In contrast, the process of Figure 4 does not describe a process in which the tank is drained. The Examiner has not stated a *prima facie* case that either of the cited references disclose, or make obvious, draining the tank at least once per week. The Examiner in fact does not discuss the

requirement in claim 16 that the tank be drained at least once a week at all. In contrast, Smith (at column 10, lines 59-68) states that the prior art process, involving draining the tank of water, refilling it with cleaning solution, draining the tank again, and then refilling it, is highly undesirable. A highly undesirable process would not be performed frequently. As mentioned above, Del Vecchio describes a similar process (called "deep cleaning") at column 10, lines 8 to 15, and in more detail at column 11, line 47 to column 12, line 65. Del Vecchio describes the deep cleaning process as having a duration of "preferably several hours" (column 12, lines 16-20) and being performed more or less "once per month" (column 12, lines 20-23). The Appellants submit that it would not be obvious to perform a cleaning process that takes several hours once per week if once per month is adequate. Further arguments in relation to Del Vecchio are given in the section of this brief regarding the obviousness rejection of claim 26 over Del Vecchio alone. The Appellants submit that the rejection fails to establish that all elements of claim 16, combined as claimed, were obvious and so fails to establish that claim 16 is obvious.

The Appellants submit that the dependent claims are all allowable for at least the reasons given in relation to their parent claim 26. However, additional reasons why the dependent claims are allowable are given below.

Claim 27

Claim 27 requires, among other things, that the cycle of step (i) of claim 26, which includes a step of draining the tank of the retentate, be repeated at least once a day. The Examiner cites figure 6 and column 13, lines 50-57 of Smith. These references do not describe a process having any step of draining a tank at all, much less at least once a day. The undesirability of the prior art process involving draining a tank stated in Smith, and the several hour duration of the deep cleaning process in Del Vecchio, all suggest that it would not be obvious to have a step of draining a tank daily.

Claim 28

Claim 28 requires a further step of performing recovery cleanings to increase the permeability of the membranes from time to time and states that the cleaning events of claim 26 are performed between these recovery cleanings to reduce the rate of a decline in the permeability of the membranes.

The Examiner refers to column 13, lines 50-57, column 18, lines 5-12, column 10, lines 64-68 and column 11, lines 22-30. These references do not describe any process having the steps of claim 28. The Examiner then refers to various generic factors (discussed in either Smith or Del Vecchio) that might influence the need for cleaning and argue that it would be obvious to optimize "the cleaning cycles" in response to these factors. Such an argument fails to raise a *prima facie* case for obviousness, firstly, because the cited references do not disclose or make obvious any process having all of the elements of claim 28 performed according to any parameters. Secondly, the Examiner has not established that any optimization of the process in the cited references would lead towards any part of claim 28. There is no teaching in either reference that cleaning steps involving a chemical cleaner should be performed in a way that reduces a rate of decline in permeability between recovery cleanings that increase membrane permeability. Both Smith and Del Vecchio teach that any chemical cleaning should be effective to create a large increase in permeability. Smith, for example at column 12, lines 46 to 55, states that the goal of each cleaning is to provide a restored flux equal to at least 70% of an initial stable flux. Optimization according to this goal would not lead to providing the steps of claim 26, with their weekly undesirable tank draining steps, in a manner intentionally designed to merely reduce the rate of permeability decline and further having recovery cleaning steps to restore membrane permeability form time to time. In Del Vecchio, the only chemical cleaning is the "deep cleaning" which is a time intensive step, performed infrequently to eliminate or reduce biomass that may have

accumulated on the membranes during operation (column 12, lines 11 – 16). While Del Vecchio suggests (column 12, lines 20-23) that the monthly period between deep cleanings might vary depending on the needs of the system, nothing in either reference suggests that the frequency might be varied to once a week or more, that the deep cleanings be intentionally be made to produce a rate of decline in permeability, and that additional recovery cleaning steps be added to increase the permeability from time to time.

Claims 29 and 30

Regarding claims 29 and 30, none of the Examiner's references to Smith describe a weekly CT value (sum of the products of chemical concentration and duration of contact with the membranes for all steps of wetting the membranes with cleaning chemicals conducted in a week). The reference at column 11, lines 30-55 of Smith '479 describes a maximum duration of a cleaning event. The references to the table and column 15 lines 34-36 give sample concentrations without suggesting an appropriate duration to be used with such a concentration. None of these references discuss how many cleanings of any particular duration or concentration would be done in a week. Finally the Examiner argues that, "these ranges are optimizable depending on the water quality and membrane flow rates". The Examiner has not provided any evidence to support this statement whatsoever. Smith does not disclose that CT is a variable that achieves a recognized result in any process, much less a process as described in claim 26. Smith also does not teach that determining an optimal or workable range of CT should be part of routine experimentation. Accordingly, the Examiner has not established a *prima facie* case that weekly CT has been recognized as a result effective variable in the context of a process as described in claim 26, or even if were a result effective variable, that routine experimentation or optimization of the process in Smith would obviously produce the claimed weekly CT in the context of a process as described in claim 26.

Accordingly, the Examiner has not established the requirements of a *prima facie* case of obviousness under the doctrine of result effective variables (see MPEP 2144.05 II A and B).

Claim 31

Regarding claim 31, which describes recovery cleanings at least 1 month apart from each other, the Examiner refers to Figure 4 of Smith. However, Figure 4 describes an experiment lasting only about 16 days. The Examiner cites col. 1, lines 18-22 but this passage only makes a vague reference to membranes being "periodically cleaned", and does not relate to the process of the Smith invention but to a prior art process having "inside-out" flow whereas Smith relates to "outside-in flow" (column 1, lines 23-25). The Examiner then asserts that discovery of an optimum value of a result effective variable in a known process is ordinarily within the skill of the art. However, claim 31 depends on claim 28 which, for the reasons given above, is not a known process. Further, the Examiner has not provided any evidence that the time between recovery cleanings is known to be a result effective variable. The standards of MPEP 2144.05 II A and B are not met.

Claim 32

The Appellants rely, for the purposes of the rejection of this claim on this ground in this appeal, only on the arguments made in relation to claims 26 and 28.

Claim 33

Regarding claim 33, the Examiner has not provided prima facie evidence that Smith discloses a process as in claim 26, with a weekly CT in the range specified in claim 29 wherein the steps of part (f) of claim 26 are performed at regular intervals and each have about the same product of concentration and duration. The Examiner merely references the "abstract and figures of Smith" which does not disclose a combination having all of the elements of the claims.

Claims 34

Claim 34 states that the membranes are backwashed with permeate after being wetted with a cleaning chemical and before starting a new cycle. The Examiner's reference to Smith column 12, lines 26-68 fails to provide the elements of this claim. Similarly, the reference to column 12 of Del Vecchio fails to provide the elements of this claim for the reasons given later in this brief in relation to the rejection of claim 34 over Del Vecchio alone.

Claims 35 and 36

The Appellants rely, for the purposes of the rejections of these claims on this ground in this appeal, only on the arguments made in relation to claims 26.

IV. The Section 103(a) Rejection of Claims 26-28, 31 and 34-36 over Del Vecchio

Claims 26-28, 31 and 34-36 were rejected as being obvious over Del Vecchio applied alone. The Appellants submit in brief that the cited reference fails to provide or make obvious all elements of these claims combined and arranged as in the claims.

Claim 26

Claim 26 requires, among other things, wetting membranes with a cleaning chemical, while or after draining a tank of retentate, at least once a week. In relation to this element of the claim, the Examiner argues (at page 10 of the Office Action), that the once a week frequency would be obvious, or could be optimized for the process conditions, based on the teaching at column 12, lines 12-30 of Del Vecchio. Column 12, lines 12-30 are part of the description of a multi-step "deep cleaning" process which occurs during an interruption of normal operation (column 9, lines 48-56). The relevant part (column 12, lines 12-23) of the cited paragraph is reproduced below:

> The membrane cartridge 216 is then "soaked" in the cleaning solution for a predetermined period of time in order to eliminate or reduce the amount of bio-mass that may have accumulated on the surfaces of the fibers 268 of the membrane cartridge 216. Although various durations may be selected depending on the particular constituents of the substrate and biomass and other factors, the duration of the cleaning operation is preferably several hours and preferably as long as four hours or longer. Such "deep cleaning" may be advantageously performed once per month of normal operation or at more or less frequent intervals depending on the needs of the system and the rate at which a bio-film is generated on the fibers.

The prior decision of February 16, 2007 on Appeal 2006-2492 held, at page 7, that the passage above did not expressly or inherently describe a frequency of at least once a week. Since the claimed frequency is not described in the claim, the Examiner has the burden of establishing that the claimed range is somehow obvious. To do so, pursuant to MPEP 2144.05 II, the Examiner would need to establish, firstly, that "deep cleaning" frequency was recognized as a variable which achieves a recognized result and, secondly, that optimization within the conditions in Del Vecchio or by routine experimentation would result in the claimed range. The Appellants submit that the Examiner has not satisfied either part of his burden.

Regarding the first part of the test, Del Vecchio does not teach that varying "deep cleaning" frequency achieves any particular result. On the contrary, to the extent that "deep cleaning" frequency might be varied, it is varied in response to the rate at which a bio-film is generated on the fibers. This amounts to little more than a teaching that when the fibers become unacceptably fouled, they should be cleaned. Del Vecchio does not teach any improvement to the overall water treatment process that can be achieved by altering "deep cleaning" frequency. In contrast, the Appellants' application as filed, at page 17 line 21 to page 18, line 5,

describe a process which is implemented before the membranes foul significantly, to reduce the frequency of intense cleaning procedures such as the "deep cleaning" in Del Vecchio. The Appellants' parameters are chosen to achieve a defined goal, a reduced rate of decline in membrane permeability between recovery cleanings, whereas Del Vecchio teaches no goal to be achieved by proactively modifying "deep cleaning" frequency.

Regarding the second part of the test, optimization or routine experimentation around the conditions in Del Vecchio will not obviously lead to a frequency of at least once per week. The starting point for experimentation in Del Vecchio is once a month. Since the "deep cleaning" process interrupts normal operation for several hours, there is a disincentive to experiment with ranges much shorter than once a month. A person skilled in the art would seek to interrupt the process as infrequently as possible. Further, Del Vecchio teaches that the "deep cleaning" process need only be performed in response to significant fouling. There is no teaching towards the use of "deep cleaning" procedures before they are actually required. In contrast, the Appellants teach the use of the claim 26 process to clean the membranes with chemical solutions before the membranes foul significantly. Claim 26 is accordingly outside any obvious range of experimentation according to Del Vecchio's teachings.

The Appellants submit that the dependent claims are all allowable for at least the reasons given in relation to their parent claim 26. However, additional reasons why the dependent claims are allowable are given below.

Claim 27

Claim 27 states, among other things, that a cycle including a step of draining a tank, and a step of wetting membranes with a cleaning chemical are repeated at least once a day. The Examiner again recites the passage described above to a "deep cleaning" process (column 12, lines 19-20) performed once a month, more

or less. The Examiner further cites a "pulsed cleaning" described at column 10, lines 4-8. The complete description of pulsed cleaning is provided at column 9, line 63 to column 10, line 8. "Pulsed cleaning" is not the same as "deep cleaning" and does not involve draining a tank. Pulsed cleaning also involves permeate (column 9, line 64), which is filtered water (column 4, lines 34-38) not a cleaning chemical. Accordingly, neither the "deep cleaning" nor the "pulsed cleaning" involve contacting the membranes with a cleaning chemical and draining a tank at least once a day.

Claims 28 and 31

Claims 28 and 31 describe additional steps of performing recovery cleanings (at a frequency of once a month or less in claim 31) to increase membrane permeability and that the steps of claim 26 occur between these recovery cleanings to reduce a rate of decline in membrane permeability between recovery cleanings. To provide the recovery cleaning step, the Examiner refers to "deep cleaning" or a modified version of "deep cleaning" described at column 12, lines 30-40. However, this "deep cleaning" is the same process that the Examiner alleged provided step (f) of claim 16. The same disclosure cannot simultaneously anticipate both the cleaning steps of claim 16 and the additional recovery cleaning steps of claims 28 and 31.

The Examiner suggests that recovery cleaning is that which is described in paragraph 8 of the Appellants' pre grant publication, or page 3, lines 2-6 of the application as filed. Recovery cleaning is actually described from page 2, line 21 to page 3, line 11 as cleaning instituted to substantially restore the permeability of membranes, for example membranes that have been in use for at least a couple of weeks and have fouled to an unacceptable level. The portion of the Appellants application cited by the Examiner is merely part of the description of one type of recovery cleaning. The Appellants do not dispute that the "deep cleanings" in Del Vechhio could be recovery cleanings as defined in claims 28 or

31. However, the "deep cleanings" cannot simultaneously be the recovery cleanings recited in claims 28 and 31 and the steps of claim 26 performed to reduce the rate of a decline in permeability between recovery cleanings.

In relation to the frequency of recovery cleanings in claim 31 ("wherein the recovery cleanings are performed at least 1 month apart from each other"), the Examiner argues that the frequency of the "deep cleanings" in Del Vecchio could be at least once within the teachings of Del Vecchio. That may be so, but it is not possible for the "deep cleanings" to be simultaneously performed at least once a month apart from each other as required for the recovery cleanings in claim 31, and at least once a week, as required to satisfy claim 26. Accordingly, Del Vecchio can not make claims 28 or 31 obvious.

Claim 34

The Examiner's reference to column 12, lines 30-40, describes pulsed cleaning (backwashing) during the deep cleaning. In contrast, claim 34 requires backwashing after the claim 26, step (f) cleaning step. The Examiner however argues that it would be obvious to also backwash the membranes again after draining the tank of chemical cleaner because Del Vecchio teaches that cleaning chemical in the system could be consumed or neutralized by the wastewater. That teaching is provided at column 12, lines 49-56. However, as stated in that passage, cleaning solution might be degraded in the cleaning step, not by wastewater. The teaching is that in these cases the step of draining the tank for the second time of cleaning chemicals can be omitted. This has nothing to do with backwashing the membranes after the claim 26, step (f) cleaning step.

Claims 35 and 36

For the purpose of this rejection on this appeal, the Appellants rely only on their submissions above that Del Vecchio does not provide or make obvious all elements of claim 26.

V. The Section 103(a) Rejection of Claims 29, 30, 32 and 33 over Del Vecchio in view of Smith

Claims 29, 30, 32 and 33 (all pending claims not rejected under Del Vecchio alone) were rejected as being obvious over Del Vecchio as applied to claim 26 in the rejection over Del Vecchio alone, but further in view of Smith. The Applicants' arguments in brief are that Del Vecchio does not make the elements of claim 26 obvious for the reasons given above, that Smith does not provide the additional elements of claims 29, 30, 32 or 33 for the reasons given in relation to the obviousness rejections based on Smith in view of Del Vecchio, and that there is no teaching towards the combination of these two references that would produce the claimed process.

Claims 29 and 30

The Examiner provides no discussion as to where the additional elements of claims 29 and 30 are provided in either reference or evidence of a teaching towards a combination of references. Accordingly, the Examiner has failed to provide a prima facie case of obviousness. The Applicants further repeat and rely on their comments in relation to claims 29 and 30 in traversing the rejection of these claims for obviousness over Smith in view of Del Vecchio.

Claim 32

For the purposes of this appeal, the Appellants rely only on their comments in relation to claims 26 and 28 in response to the rejection of this claim on this ground.

Claim 33

For the purposes of this appeal, the Appellants rely only on their comments in relation to claims 26 and 29 is response to the rejection of this claim on this ground but note that the Examiner's rejection is based only on Del Vecchio.

Summary

For the foregoing reasons, the Appellants believe that the Examiner's rejections of claims 26-36 were erroneous and reversal of his decision is respectfully requested.

Respectfully submitted,

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CLAIMS APPENDIX

26. A process for filtering water containing solids with membranes in a tank comprising the steps of:

a) filling the tank with a feed water to be filtered to immerse the membranes;

b) creating a transmembrane pressure between a permeate side and a retentate side of the membranes, the retentate side of the membranes being in contact with the water in the tank at ambient pressure, the permeate side being subject to a negative pressure relative to the pressure of the water in the tank fluidly connected to a filtered permeate outlet, to generate a filtered permeate at the permeate outlet and a retentate in the tank;

c) aerating the membranes to dislodge solids from the membranes;

d) backwashing the membranes; and,

e) draining the tank of the retentate;

wherein

i) the steps above are performed in repeated cycles; and,

ii) the steps of backwashing the membranes and draining the tank in a cycle may be performed either before the other or partially or substantially simultaneously; and,

f) wetting the membranes at least once per week with a cleaning chemical having a selected concentration for a selected duration after performing step (b) in a first cycle and after or while performing step (e) in the first cycle, without returning to step (b) in the first cycle and before starting a subsequent cycle.

27. The process of claim 26 wherein the repeated cycles of part (i) of claim 26 are repeated at least once a day and step (f) is repeated between once a day and once per cycle of part (i) of claim 26.

28. The process of claim 26 further comprising the steps of performing recovery cleanings from time to time to increase the permeability of the membranes wherein the steps of claim 26 are performed between the recovery cleanings and reduce the rate of a decline in permeability of the membranes between the recovery cleanings.

29. The process of claim 26 wherein the sum of the products of the selected concentration and selected duration of part (f) of claim 26 is between 2,000 min•mg/l and 20,000 min•mg/l per week over a period of at least 1 month when NaOCl is the cleaning chemical or an equivalent product of concentration and time of another cleaning chemical.

30. The process of claim 29 wherein the sum of the products of the selected concentration and selected duration of part (f) of claim 26 is between 5,000 min•mg/l and 10,000 min•mg/l per week over a period of at least one month when NaOCl is the cleaning chemical or an equivalent product of concentration and time of another cleaning chemical.

31. The process of claim 28 wherein the recovery cleanings are performed at least 1 month apart from each other.

32. The process of claim 28 wherein the filtered permeate generated at the permeate outlet is intended for use as drinking water and the cleaning chemical comprises an oxidant.

33. The method of claim 29 wherein the steps of part (f) of claim 26 are performed at regular intervals and each have about the same product of selected concentration and selected duration.

34. The method of claim 26 wherein the membranes are backwashed with permeate in the first cycle after step (f) of claim 26 in the first cycle and before starting the subsequent cycle.

35. The method of claim 26 wherein step (f) of claim 26 further comprises the steps of flowing water to the permeate side of the membranes and mixing a cleaning chemical into the flowing water.

36. The method of claim 26 wherein the membranes are hollow fibre porous membranes.

EVIDENCE APPENDIX

1. U.S. Patent No. 5,403,479 to Smith et al.

2. U.S. Patent No. 6,331,251 to Del Vecchio et al.

US005403479A

United States Patent [19]

Smith et al.

[11] Patent Number: 5,403,479

[45] Date of Patent: Apr. 4, 1995

[54] **IN SITU CLEANING SYSTEM FOR FOULED MEMBRANES**

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[73] Assignee: **Zenon Environmental Inc.**, Ontario, Canada

[21] Appl. No.: **170,053**

[22] Filed: **Dec. 20, 1993**

[51] **Int. Cl.**[6] **B01D 63/00**

[52] **U.S. Cl.** **210/321.69**; 210/321.8; 210/321.89; 210/257.2; 210/195.2; 210/636

[58] **Field of Search** 210/636, 108, 195.2, 210/257.2, 791, 321.69, 333.9, 106, 321.8, 321.89, 321.87, 321.88, 500.23

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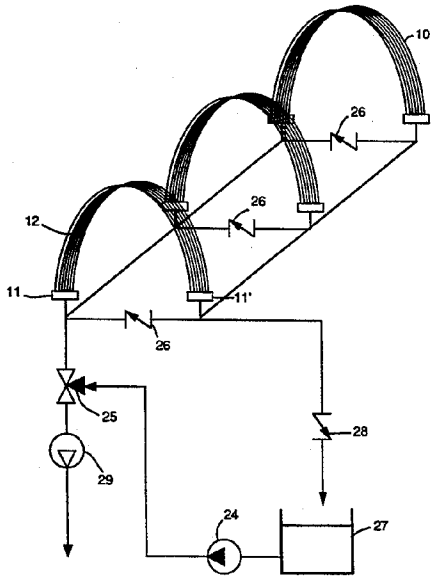
Primary Examiner—Robert A. Dawson
Assistant Examiner—Ana M. Fortuna
Attorney, Agent, or Firm—Alfred D. Lobo

[57] **ABSTRACT**

A method and cleaning system is disclosed for cleaning the outer surface of a fouled microfiltration (MF) or ultrafiltration (UF) semipermeable hollow fiber membrane after its initial stable transmembrane flux has been decreased to an unacceptably low level. The method is specifically applicable to any fiber used to withdraw purified water from dirty water, particularly water containing organic matter including beneficial bacteria and/or undesirable inorganic salts, where the viability of the bacteria population is to be maintained. The membrane is cleaned by flowing a cleaning fluid, preferably a biocidal oxidative electrolyte having an oxidizing anion and an associated cation through the clean, permeate-side of the membrane, at low pressure no more than the bubble pressure breakthrough, usually <300 kPa (30 psig) for a MF or UF fiber. Such low pressure is sufficient to diffuse the electrolyte through both, the pores of the membrane and a fouling film which typically includes a biofilm accumulated on the fibers' outer surface, but not enough electrolyte flows through the membrane to kill numerically more than 20% of the living bacteria in the dirty water. This limitation can be met only if the cleaning period is brief. This period is only long enough to oxidize organic matter within the pores and kill essentially all bacteria in the biofilm. Preferably less than 5% of the bacteria population is decimated. As diffusion takes place, pores are again opened, both in the wall of the fiber and through the biofilm, and when the fibers are returned to normal operation, the restored flux is equal to at least 70% of the initial stable flux.

2 Claims, 8 Drawing Sheets



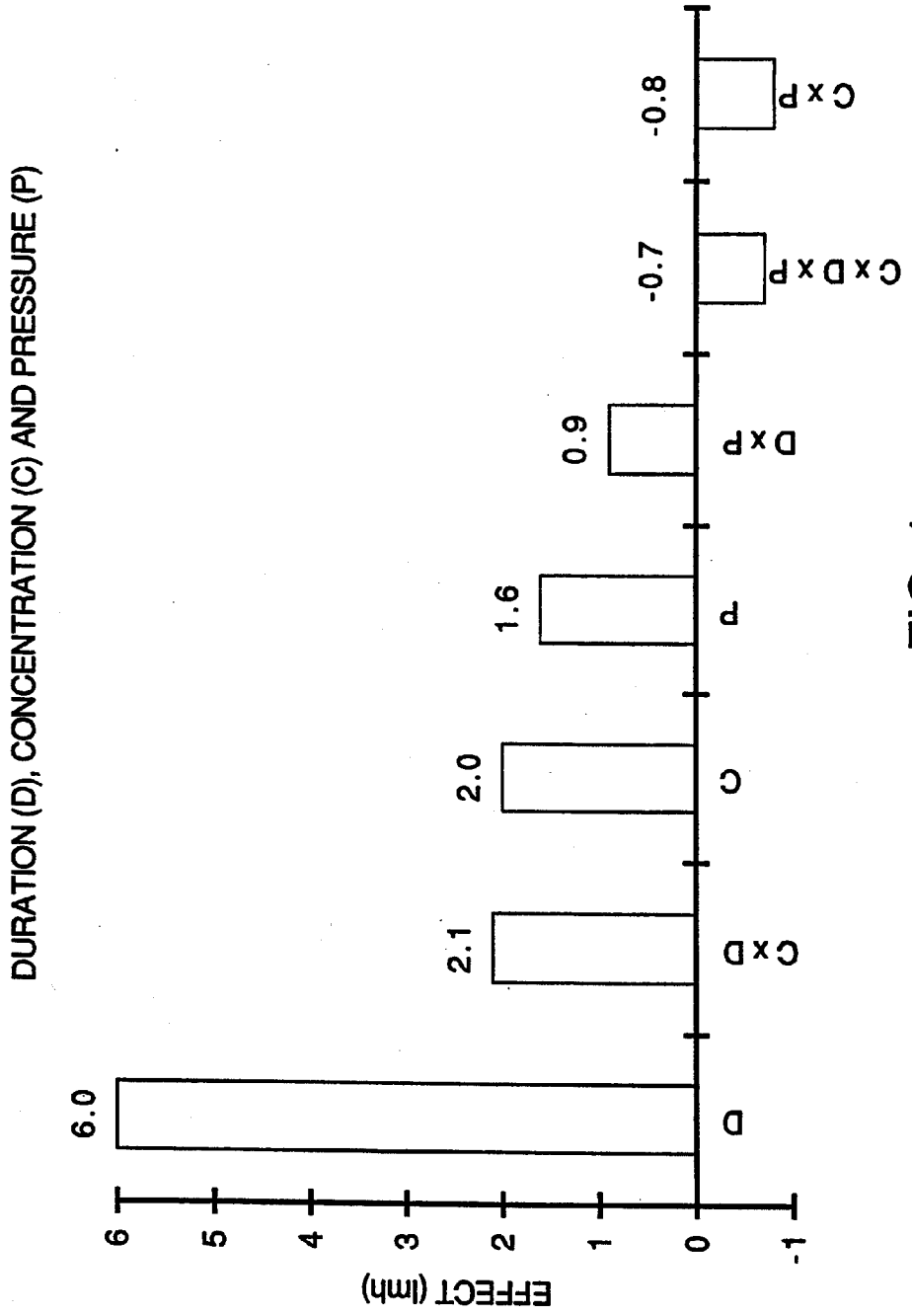

DURATION (D), CONCENTRATION (C) AND PRESSURE (P)
EFFECT (lmh)
6
5
4
3
2
1
0
-1
6.0
2.1
2.0
1.6
0.9
-0.7
-0.8
D
C x D
C
P
D x P
C x D x P
C x P

FIG. 1

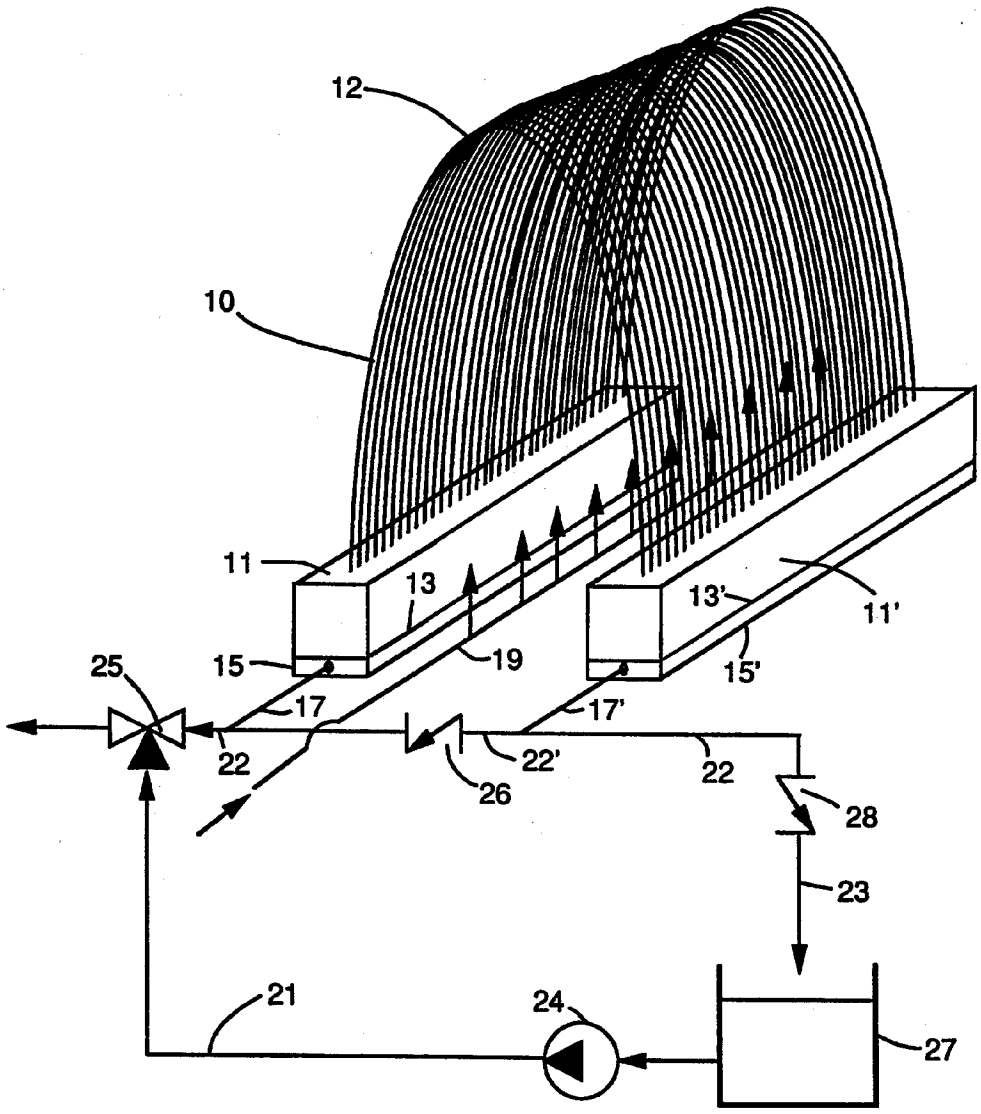


FIG. 2

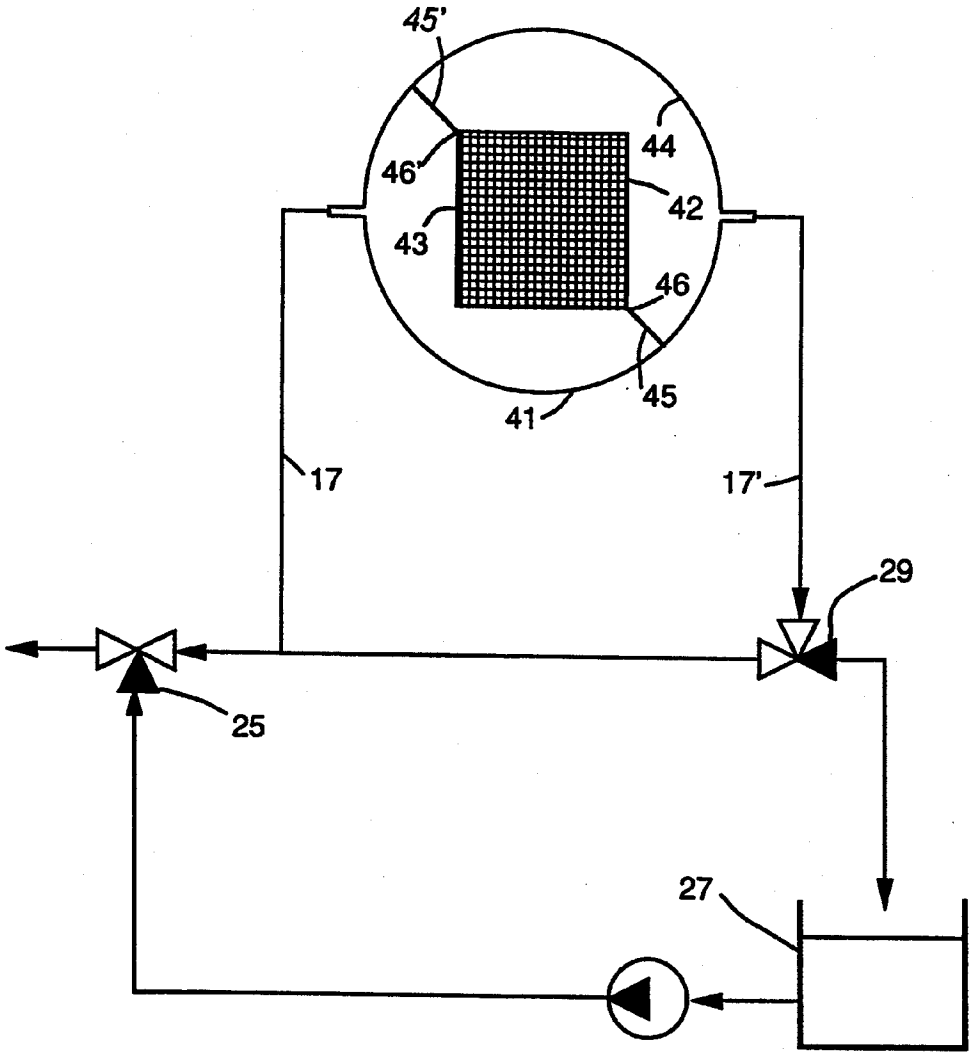


FIG. 3

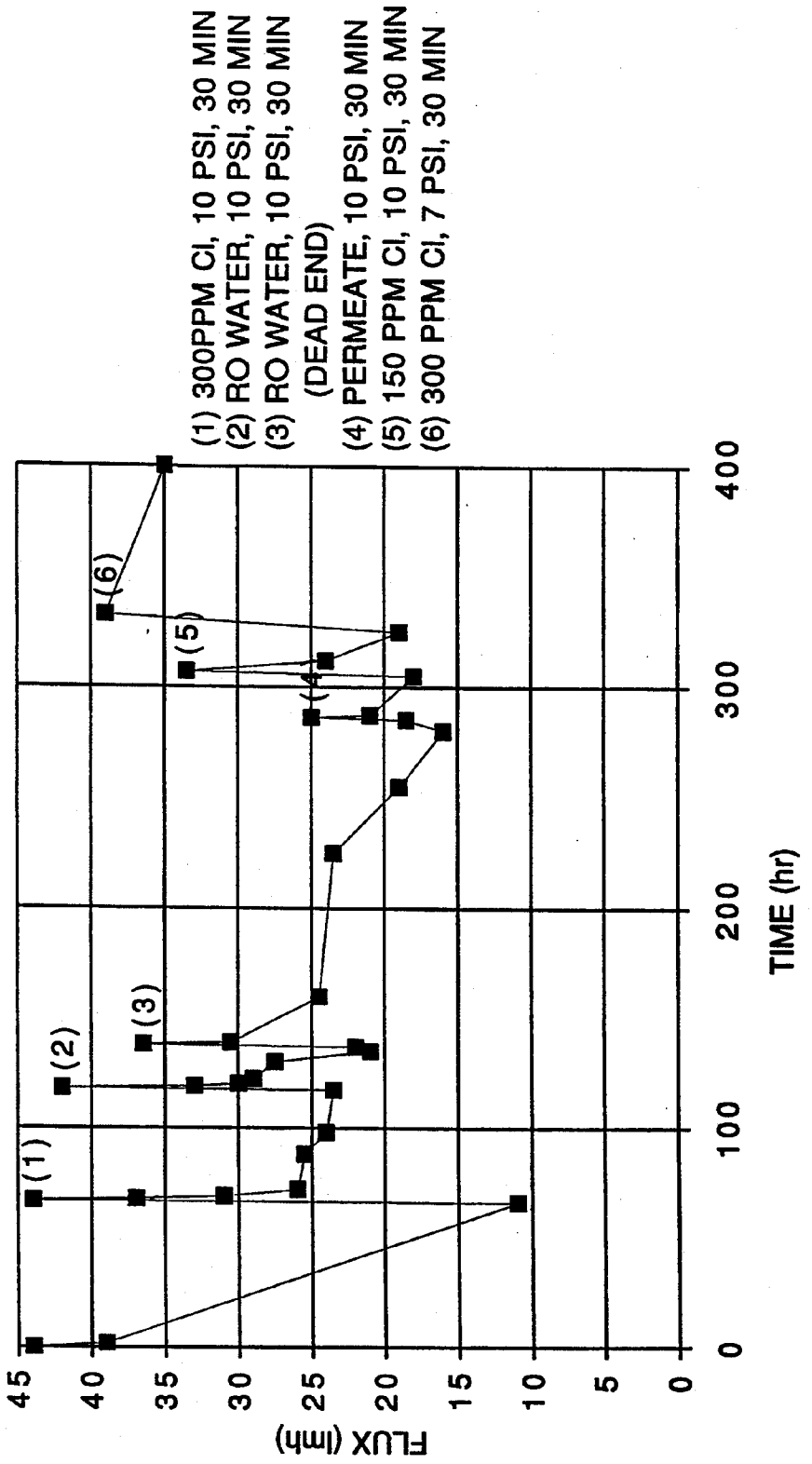

(1) 300PPM Cl, 10 PSI, 30 MIN
(2) RO WATER, 10 PSI, 30 MIN
(3) RO WATER, 10 PSI, 30 MIN
(DEAD END)
(4) PERMEATE, 10 PSI, 30 MIN
(5) 150 PPM Cl, 10 PSI, 30 MIN
(6) 300 PPM Cl, 7 PSI, 30 MIN
FLUX (lmh)
45
40
35
30
25
20
15
10
5
0
0
100
200
300
400
TIME (hr)
(1)
(2)
(3)
(4)
(5)
(6)

FIG. 4

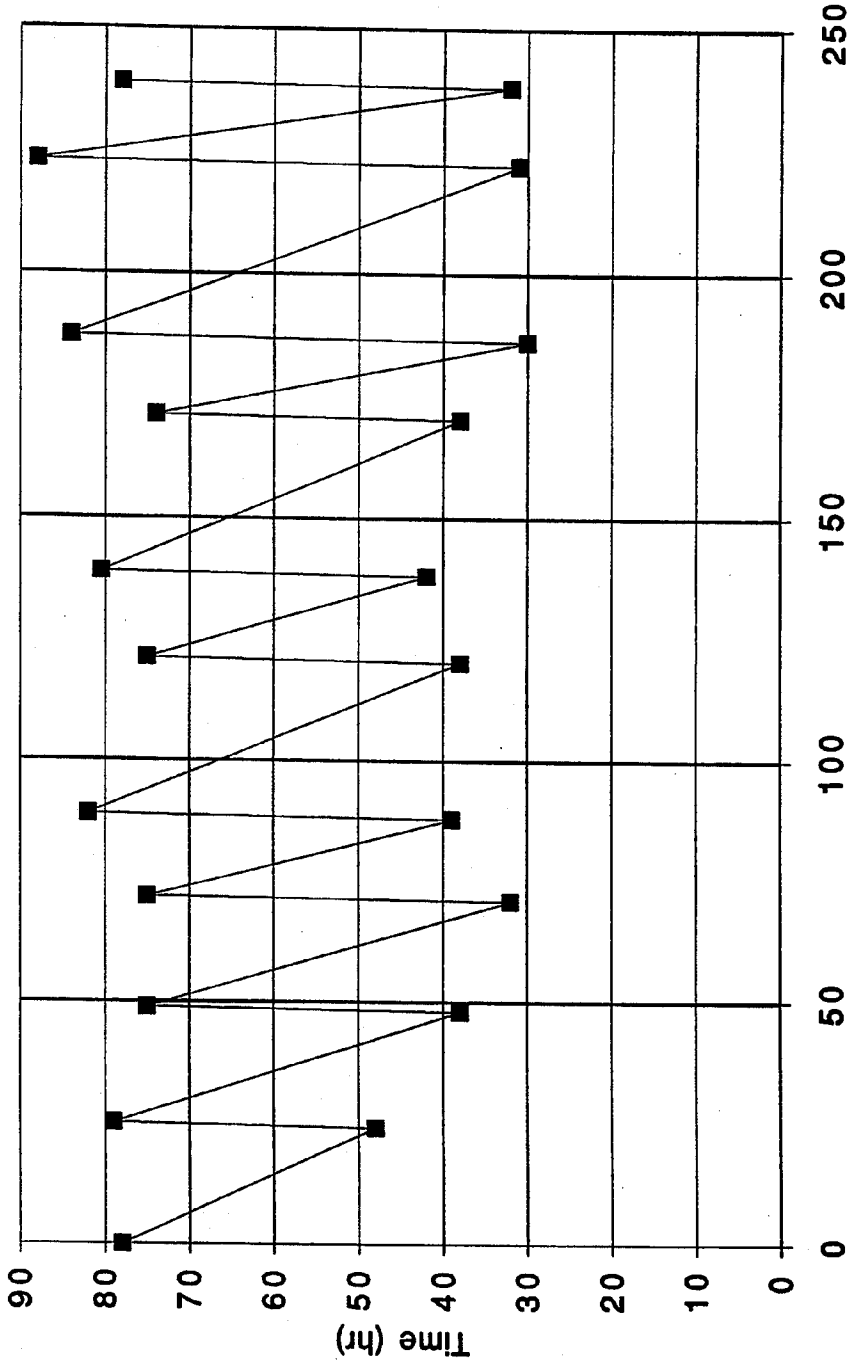


FIG.5

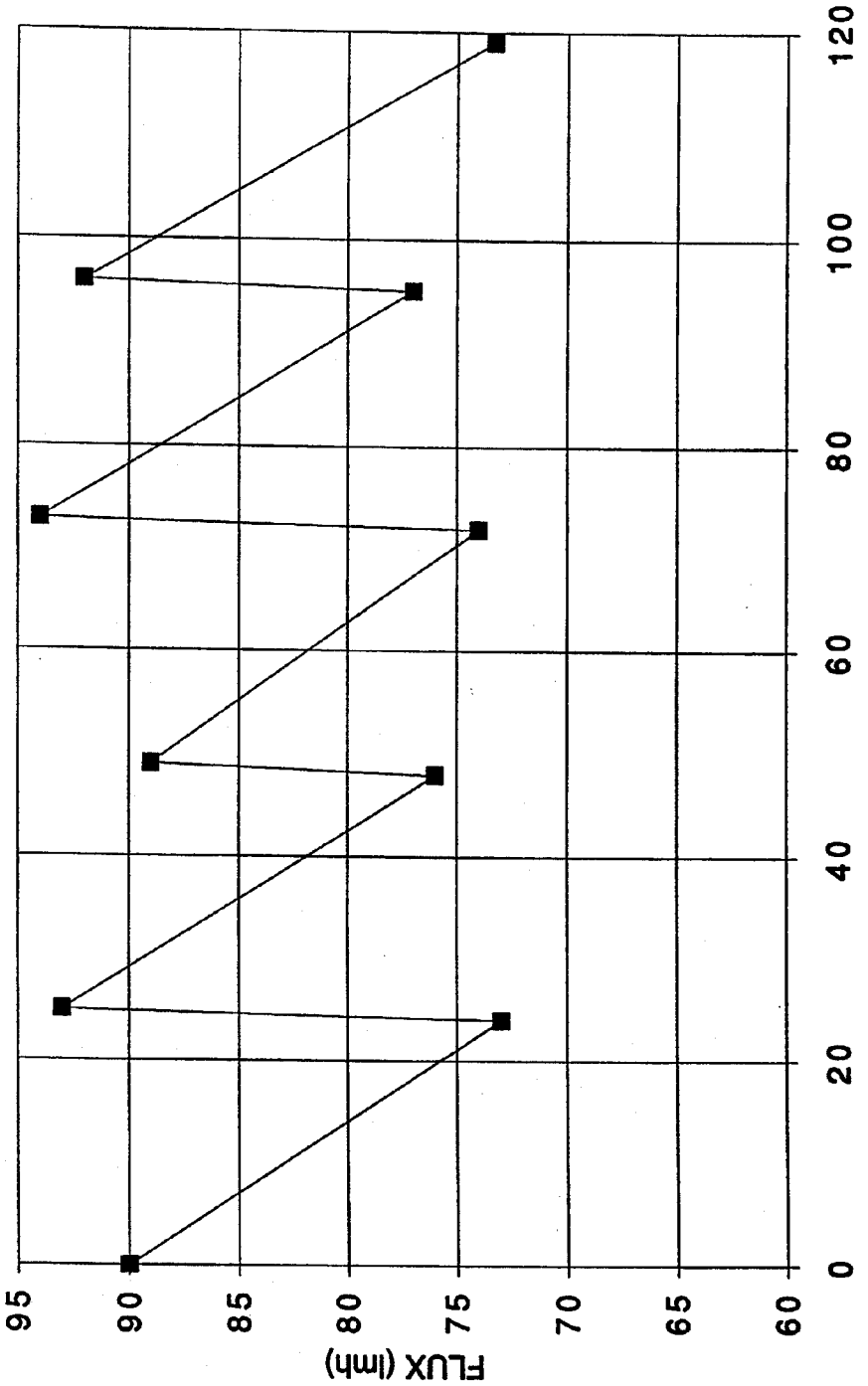


FIG.6

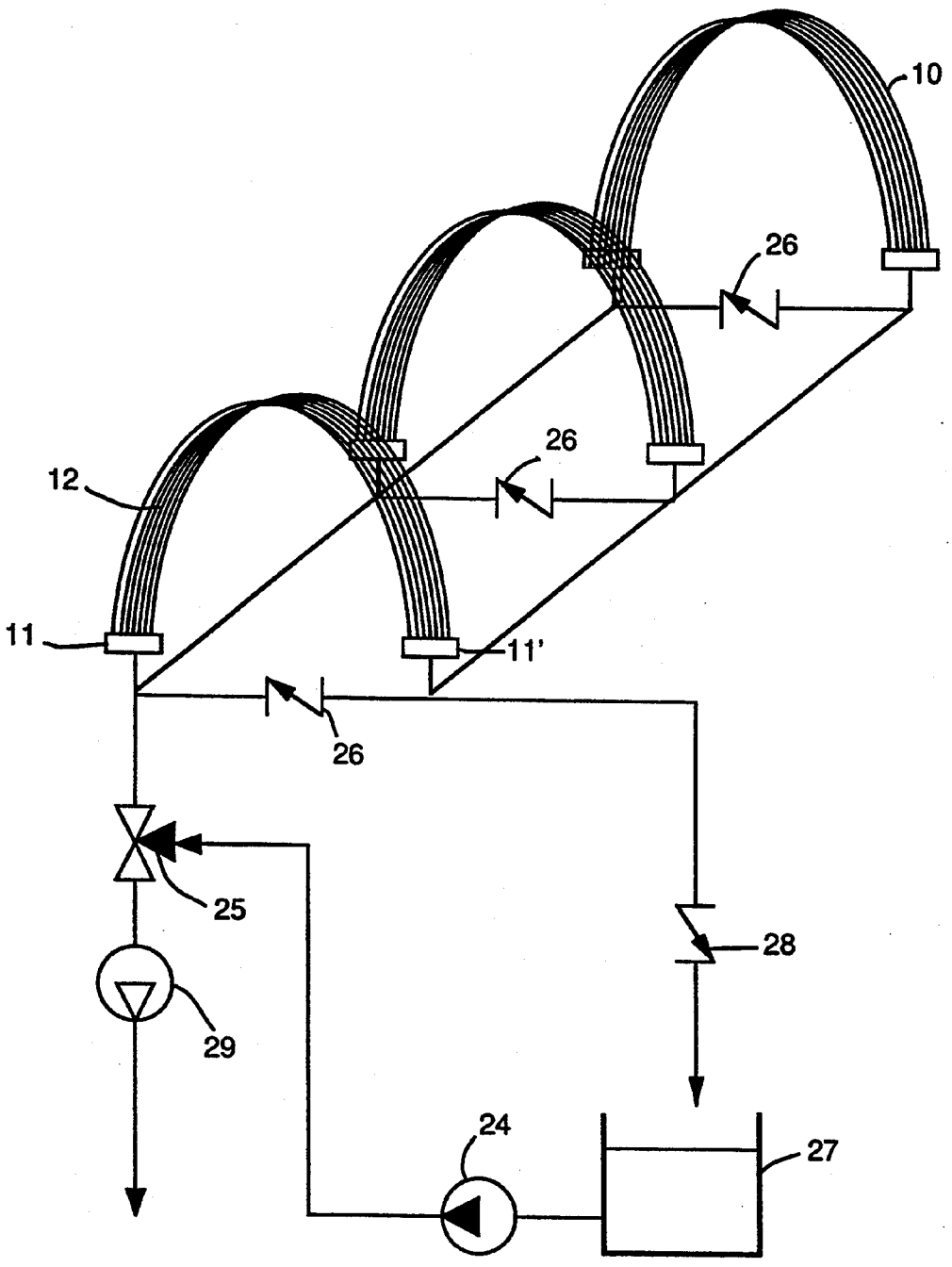


FIG. 7

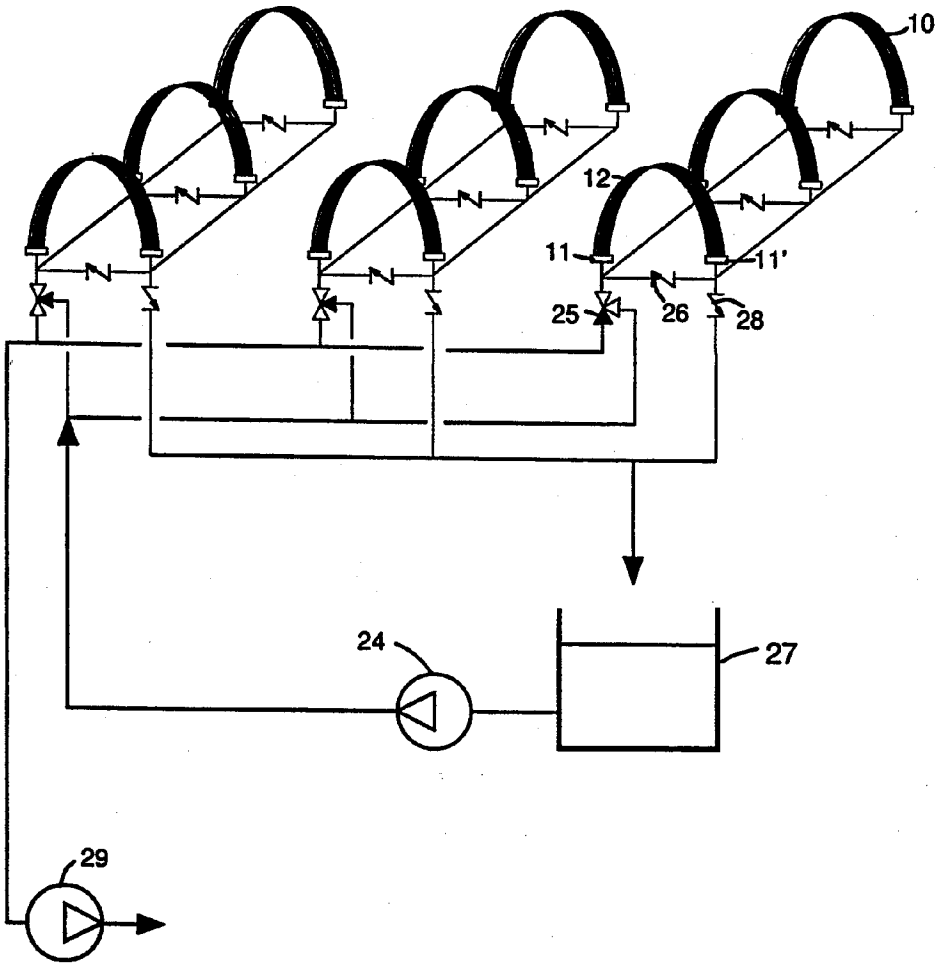


FIG. 8

IN SITU CLEANING SYSTEM FOR FOULED MEMBRANES

BACKGROUND OF THE INVENTION

This invention relates to a cleaning system for substantially restoring transmembrane flux (hereafter "flux" for brevity), measured as liters of permeate per square meter of membrane surface per hour ($L/m^2.hr$ or "LMH"), in fouled, porous/semipermeable microfiltration (MF) or ultrafiltration (UF) membranes in a membrane device (module) used to recover purified water from contaminated or "dirty" water in feedstream, without draining the feed (substrate), hence referred to as an "in situ cleaning" method. A MF or UF membrane is generally used to separate one liquid, usually water, from water containing various forms of undesirable matter, some in solution and some not. Such a membrane device which is to be periodically cleaned, usually operates in "inside-out flow" in which the inner surfaces of the membranes are exposed to the feedstream of "dirty" water from which purified water is to be separated. In contrast, this invention relates to hollow fiber membranes ("fibers" for brevity) which typically operate in "outside-in" flow. By hollow fiber membranes we refer to membranes having an inside diameter (i.d) in the range from about 0.2 mm to 4.0 mm, with a wall thickness which corresponds to a particular diameter, the outside diameter (o.d.) usually being in the range from about 0.3 mm for the smallest fibers to about 6 mm for the largest.

The term "dirty" water is used herein, in a generic sense to refer to any poor quality aqueous, or predominantly aqueous solution, suspension, dispersion or emulsion. Purified water is extracted from the dirty water with a desirably high flux despite the membrane being covered, in about 8 hr or less, with a "fouling film" deposited by "foulant(s)" in the substrate. This formation of the film is also referred to as concentration polarization which is unavoidable in practice. A foulant film formed in an aqueous medium rich in microorganisms ("biomass") is termed a "biofilm", and the fouling phenomenon is referred to as "biofouling". By "rich in microorganisms" we refer to a cell count in excess of 5000 CFU/ml (colony forming units/ml). Other types of fouling occur in other applications, for example in the purification of water containing multivalent cations in the form of Ca Mg Si Fe and Mn salts (carbonates, oxides, chlorides and the like). When the fouling film decreases the desirably high flux, the membrane is cleaned to substantially restore the flux to a desirable level.

The cleaning method of this invention is particularly directed to cleaning fibers, rather than tubular membranes or spiral wound membranes. Fibers are used in a module, either in an array or in a bundle, deployed directly in a substrate without being enclosed; or, the array may be appropriately held within a shell. With fibers enclosed in a module, feed flowed through the shell side and over the outer surfaces of a multiplicity of fibers held therewithin, and emerging from the shell, is referred to as retentate or, more preferably, concentrate; and, liquid which is separated by, and flows through the microporous membrane into the lumens of the fibers is referred to as "tiltrate", or preferably, "permeate".

Restoration of the flux is effected on the permeate side of the membrane, with a cleaning fluid, most preferably an aqueous cleaning fluid, under only enough pressure, below the bubblepoint of the fiber, which for reasons given below, is believed to provide diffusion-controlled permeation. Other mechanisms may also play a part in cleaning. For example, since the membranes used herein are of a synthetic resinous material, rather than being ceramic, they are susceptible to swelling caused by interaction with the cleaning fluid.

Diffusion-controlled flow occurs at low pressure through the walls of the membranes and out into the feed (hence referred to as "inside-out flow" of a "substantially pressureless" cleaning solution). The definition of "diffusion-controlled" permeation is that which occurs at a pressure below the "bubble-pressure breakthrough" (or "bubble-point") for a membrane, and the permeating rate "J" is measured in gm-moles/sec/cm^2. This definition is adapted from a method for measuring the pore sizes of a membrane by diffusion of air through water which fills the pores of the membrane at the "bubble-pressure breakthrough" for a membrane. Strictly, the pressure at breakthrough is measured by the force required to force one immiscible fluid through the pores of a membrane previously filled with a second immiscible fluid. (see Membrane Handbook edited by W. S. Winston Ho and Kamaalesh K. Sirkar, Chapter VII "Ultrafiltration" pg 426 Van Nostrand Reinhold, New York). This method was originally practiced by placing a water-filled membrane with air impingement from below. Bubbles of air penetrate the membrane into an overlying water layer. The largest pores open at the lowest pressure; thus, by slowly increasing the air pressure (1 bar/min) and monitoring air passage, a pore size distribution can be estimated. Though all pores are filled with water, gas will dissolve at the upstream face of the membrane, diffuse through the pores in solution and come out of solution at the lower pressures downstream of the membrane.

The value for the permeating rate is calculated from the following equation:

$$J=(N \pi d^2)/4\ (DH)\ (\Delta P/l)$$

where

J=permeating rate, gm-moles/sec/cm^2

N=pore density in number/cm^2

d=pore diameter in cm

D=diffusivity of the gas (N_2) in water at 20° C.=1.64×10^{-5} cm^2/sec

H=solubility of the gas (N_2) in water at 20° C.=6.9×10^{-7} gm moles/at/cm^3

ΔP=pressure differential (atm) across the membrane.

For example, a membrane having a pore size of 0.27 μm, a pore density of 6×10^7 pores/cm^2, and a thickness of 10^{-3} cm (10 μm) has a diffusion rate of $J/\Delta P=3.89\times10^{-10}$ gm moles/sec/atm/cm^2, and using the gas constant this becomes 0.0355 ml/min/psi/ft^2. For a 15 ft^2 cartridge tested at 30 psi the permeating rate is about 16 ml/min. (see Handbook of Separation Techniques for Chemical Engineers M. C. Porter, Appendix A).

The membrane device most preferably used for purifying non-sterile aqueous streams is a frameless array of fibers, immersed in an arbitrarily large body of water. Such a device is disclosed in U.S. Pat. No. 5,248,424 to Cote et al. An alternative is to use a device of the "shell and tube" type in which the permeate is collected from the lumens of the fibers. Such a device is disclosed in

U.S. Pat. No. 5,232,593 to Pedersen et al. A device of either type is referred to herein as a "module".

When fibers are used, only the permeate flows into the lumens, and the lumens are not fouled under normal operating conditions. Therefore there is no logical reason to consider flowing a cleaning solution through the lumens.

A typical module is used to separate one liquid from another having clusters of molecules, or larger molecules than those of the liquid to be separated; or, to separate one liquid from another liquid containing a suspension or dispersion of micron-size inorganic particles or organic particles. Such particles include bacteria both dead and alive, or, a colloidal suspension of submicron size solids, or an emulsion, from which the aqueous component is to be separated.

Depending upon whether the particles are microscopic or submicroscopic in size, the membranes may have pores ranging in size from as large as 5 μm (micrometers or microns) or as small as 50Å, and are commonly termed "semipermeable" membranes. Membranes with circumferential walls having relatively large pores are used in MF. The pores in a MF membrane range from about 300Å to 20,000Å in nominal diameter; and those in a UF membrane, from about 50Å to about 1,000Å (0.1 μm).

Of particular interest herein is the separation of purified water from "dirty" water containing undesirable metal oxides, carbonates, etc. and/or a live biomass, or a non-sterile organic or inorganic "floe", the purified water passing through the walls of a semipermeable membrane into the "permeate side" of tube and fiber membranes (outside-in flow) in the module.

The fouling film is a thin continuous layer which develops on the surface of the membrane within the first 0.25–3 hr, generally no more than about 8, after the membrane is placed in operating service. Presence of the film is inferred from concentration of foulant in the substrate feed. Such concentration may be measured as the cell count in the water phase, or the concentration of metal salts, and is judged in terms of how much performance (flux) has dropped below target. The target flux is normally the initial stable flux obtained in the 9th or 10th hour, but often in the 5th or 6th, after a new membrane is contacted with dirty water. A biofilm typically comprises cells, both dead and alive, cell debris and extracellular polymer substances (EPS), with the EPS accounting for a substantial portion of the biofilm's dry mass. Wet biofilm may contain up to 95% or more of water.

In the aforementioned filtrations with membranes, the phenomenon of microdroplets of emulsifiable organic liquids, hydrocolloids and solute particles rejected by the membrane, tend to form a viscous and gelatine-like "fouling layer" which becomes part of the fouling film on the membrane even if there are no bacteria in the suspension, and there usually are. Thus, in addition to the resistance to flow of permeate due to the physical properties of the membrane, and, the boundary layer and biofilm formed under the conditions of its environment, there is the additional resistance due to concentration polarization. Since, in addition, the fouling film attracts live bacteria and permits their build-up, the flux will rapidly drop below 10 LMH, below which one cannot usually realistically expect to operate a commercial module either effectively or profitably.

When a fouling film is formed, irrespective of the source or origin of fouling, cleaning as taught herein provides such good diffusion through whatever film is left (typically essentially none) after cleaning, that the flux, after cleaning is within 30%, preferably within 20% of the flux measured after a new and unused membrane is placed in the same service for a sufficient time to exhibit a stable, and desirably high flux after an initial soak period. This soak period varies from about 0.25 hr to 5 hr depending upon the characteristics of the bacteria and suspended solids in the dirty water. This stable, desirably high flux obtained after the initial soak period is referred to as "the initial stable flux".

Up to the present time, cleaning membranes in a module referred to removing the fouling film by applying biocides, cleaners or physically scouring the membrane when membrane geometry allows. (see article titled "Biofouling—a Biofilm Problem" by H. C. Fleming, G. Schaule and R. McDonough, in Membrane Preparation - Fouling - Emerging Processes, European Society of Membrane Science and Technology, P. Aimar and P. Aptel Editors, Vol 6, 1992). Trying to restore the permeability and flux of a membrane generally requires dealing with the film formed on the surface of the membrane, unless the "dirty" water is sterile. Fleming et al did this by adding a commercial cleaner containing non-ionic and anionic surfactants which was forced through the biofilm layer and membrane. This was followed, once the permeability was constant, by washing the cleaned membranes with clean water. Their experiments were focused on determining the relative permeability of a model biofilm with different cleaners; and their effect on the relative height of the biofilm layer (cleaners had little effect).

In further experiments, they coated a membrane with biofilm by suspending the membrane in dirty water containing bacteria and a high EPS. They then exchanged the water for a cleaning agent, and filtered it until a constant permeability was seen. They then exchanged the cleaning agent for water and again filtered until a constant permeability was seen. They followed the same protocol in each case except that one set of data was measured with stirring during filtration, and the other was with no stirring. Since in each case the cleaner was filtered until a constant permeability was seen, they were unaware of how much cleaner had been filtered at that point. Further, since there was no substrate on the "other" side of the membrane during any of their filtration steps, they clearly evinced no interest in the effect of the cleaner which they had filtered. They had no reason to evince such an interest because they failed to conceive the importance of cleaning the membrane without removing substrate.

But it was known that cells in the biofilm are more resistant to biocides than those in free suspension, and that simply killing cells had little effect with reference to restoring the flux. Still further, since Fleming et al showed that enhancement in permeability due to the application of cleaner was due to an alteration of the biofilm, not removing it, it was clear that the biofilm did not have to be removed before the flux was sufficiently restored to return to normal operation.

Since the Fleming et al experimental method was an adaptation of the prior art method in which sufficient biocide was introduced into the dirty water to kill all bacteria, a desire to save beneficial bacteria rules out either method. In the prior art, in those particular instances where the bacteria were to be saved, the tank of dirty water is drained, or the membrane removed from the tank before the biocide is applied to the outside

surfaces of the membranes. The problem is that though this method may kill all the bacteria, it does not generally remove the biofilm, and dead cells may stick to the biofilm, and usually do.

Most importantly, the prior art failed to realize that it was possible to kill most, if not essentially all, or only a controlled minor proportion of live bacteria in the fouling film, yet restore the flux. We deliberately kill only a controlled amount of the bacteria in the feed, but not so many as to be economically debilitating. This concept of deliberately sacrificing a controlled minor proportion of live bacteria on the feed side, outside the fouling film, to kill essentially all in the biofilm, is the essential basis of this invention.

With this concept it was feasible to employ the known principles of biocidal cleaning, namely that it improves performance because (i) cleaning with a biocide reduces the thickness of the biofilm, and (ii) biocides improve the permeation properties of the remaining biofouling film, though this second effect was underestimated in the prior art. It was because this second effect was targeted, that we realize the unexpected improvement provided by this invention.

Despite the findings of Fleming et al, the prior art failed to clean membranes in aqueous, non-sterile service (a) without either draining the dirty water to flush the membranes with a biocide, or, (b) without adding the biocide to the tank to kill all cells and withdrawing the biocide through the membrane until the flux was restored to a desirable level, or (c) without removing the membranes from their aqueous medium (dirty water reservoir) to clean them. Fleming et al sought to control development of the biofilm by control of the nutrient in the system, not by sacrificing up to 20 per cent of the live bacteria in the feed in the interest of flux restoration sufficient to justify return to normal operation.

Thus, to date, it has not been possible to restore the flux of a biofouled membrane without leaving an objectionable concentration of cleaning fluid (solids are unusable in lumens) in the dirty water, even if one was prepared to kill all cells. Much less was it possible, substantially to restore the flux without killing more than a controlled amount of live cells in the biomass, while killing essentially all those cells which clog the pores of the membranes.

In most membrane-separations of dirty water to recover purified water, dirty water is passed over the outer surfaces of small diameter organic or inorganic hollow fiber membranes, or through tubes, or, through a roll, and the desired liquid is recovered as a permeate which passes through the membrane and flows out the permeate-side of the membrane device. Despite the effectiveness of fibers, tubes and rolls for making a desired separation, all are so easily and badly fouled that whether such membranes can be used economically depends upon how well the fouling material ("foulant") can be quickly removed, sufficiently to restore their initial stable flux, or, to restore the flux to as close to that initial level as practical.

Because the surprisingly effective method disclosed herein for cleaning membranes uses a cleaning fluid which is most preferably a liquid biocidal oxidizing liquid, and it contacts the lumens of the fibers at low, negligibly small fluid velocity, if any, and typically at less than 1 meter/see through the lumens, the fibers are under only enough internal pressure to cause gentle permeation of the cleaning fluid through the membrane and fouling film. It is critical that the pressure for such gentle permeation be below the membrane's bubble point.

This limitation applies whether the cleaning fluid is recirculated, held stagnant, or pulsed. Because under recirculation or pulsed conditions the cleaning fluid is in laminar flow, the method is also referred to as "in situ diffusion cleaning". Such cleaning occurs even when the fluid is simply held in the fibers at no velocity, under only enough pressure to allow the fluid to diffuse through the membrane into the reservoir in which the membrane is immersed. It also occurs under low pressure (below bubble point) pulsing of the cleaning fluid to urge the fluid to take a path other than through already-clean pores, thus to improve distribution of the fluid on the permeate side, and to vary the flow pattern of distribution of fluid as the membrane's flux is restored. Since in each case there is very little flow of biocidal solution through the lumens of the fibers, and in one case (velocity=0 meter/sec) there is none, the cleaning system of this invention does not require a conventional holding tank such as used in a prior art clean-in-place system. The biocidal liquid in our system may be dispensed from a container the fluid volume of which is only slightly greater than that of the sum of the lumens of all the fibers to be cleaned simultaneously, or the sum of the bores of all the tubes, or all the spiral passages. The solution is recirculated when it returns to the container.

A further unexpected advantage is that there is no need to counteract or recover the cleaning fluid which diffuses into the feed since that amount is too small to be objectionable, typically less than 10 ppm in a reservoir of substrate, and is biooxidized at that low concentration, negating biocide build-up.

The importance of being able to maintain the surface of a membrane clean enough to make its use in a separation process practical was the primary topic of a symposium held a decade-and-a-half ago and reported in a chapter titled "Fifteen Years of Ultrafiltration" by Michaels, A. S. in Ultrafiltration Membranes and Applications edited by A. R. Cooper (American Chemical Society Symposium, Washington, 9–14 Sept. 1979, Plenum Press, New York (1980). A flux of at least 20 LMH, preferably 50 LMH, is generally desirable in commercial separations, the higher the flux, better; and as stated above, a flux below 10 LMH is generally deemed unacceptable for the purpose at hand.

The unremitting search over the past fifteen years, for better systems to provide clean working surfaces on a membrane for long period of time, at least clean enough to provide a commercially acceptable flux, has been singularly unrewarding. As a result much energy and time has been spent on the development of semipermeable membrane compositions which are less readily fouled than ones providing comparable duty in the same or an analogous service.

To clean deposits left on a membrane when dirty water (outside-in flow) contacts its outer surface, as it most often does, two cleaning methods are now generally used. A first method relies on cleaning a fouled outer surface from the outside; the second relies on cleaning the fouled outer surface from the inside. In such prior art methods the outer surface may be that of a fiber, or a tube, or a roll; the method of this invention is mainly applicable to fibers.

In the first method which relies on cleaning a fouled outer surface from the outside, the fouled surfaces are scoured, sometimes after a soaking period in a cleaning

solution made up of specific chemicals. Scouring is effected by a suspension of finely divided solids which have essentially no affinity for the membrane, the solids having a diameter larger than the largest pores in the membrane so as not to be trapped therein, the scouring action being controlled by the rate at which the suspension is flowed over the membrane surfaces.

An alternative first method uses a chemical cleaning solution to remove the solid or semi-solid matter which is deposited on the membrane's outer surface. Such a cleaning solution is aptly formulated to dissolve or chemically react with the organic or inorganic matter deposited on the membrane. A drained module may be soaked in the solution, or the solution may be recycled through the shell-side of the module until the fouling matter is chemically degraded and dislodged. It will be understood that in outside-in flow, the permeate side of the membrane (the lumens of fibers) does not get fouled because essentially no solids pass through a membrane.

To clean the exterior by exercising either of the above options, the feed must be shut off, and the module is preferably taken out of service and drained, before the chosen cleaning fluid in the appropriate concentration, is introduced in lieu of the feed. The cleaning solution is recycled over the surfaces of the membrane until they are cleaned, then discarded to drain. If a bioreactor is available, the cleaning solution is collected and gradually bled into the bioreactor where the chemicals and fouling solids are biodegraded.

Representative conventional clean-in-place systems without draining the feed are illustrated in articles titled (i) "Improved Product Rinsing Efficiency with Multitubular Ultrafiltration" by W. J. Allshouse and Masatake Fushijima, ELECTROCOAT '84, pg 14-1 to 14-13; (ii) "New Developments in Ultrafilter System Design" by Mark Rizzone, ELECTROCOAT '88, pg 11-1 to 11-39; in a reference manual titled "Koch Spirapak Electrodeposition Paint Ultrafiltration Modules" published June '89 by Koch Membrane Systems, Inc.; and in bulletins "ZPF8-Series Ultrafiltration Systems" and "LF-Series Reverse Osmosis Systems from 60 to 300 gpm" published by Zenon Environmental Systems Inc. Most recently a liquid back-washing system has been used for fibers in which permeate is withdrawn in outside-in flow. The fibers are cleaned by flowing a solution of cleaning agent through a bundle of fibers after the flow of the solution is blocked. There is no enablement of diffusion-controlled flow. No bacteria population is stated to exist in the medium, nor is there concern for maintaining the bacterial population. (see Japanese patent publication JP 4-265127A, Sept 1992).

It is important to note that reference to "back-washing" or "back-flushing" fibers in the prior art does not refer to recirculating liquid through the lumens of fibers because the pressure drop of cleaning solution through the lumens is so high. The fact that diffusion-controlled permeation did not require a substantially pressurized solution escaped notice. Because it is impractical to recirculate even a low viscosity liquid such as DI water through hollow fibers, the conventional method of "back-flushing" on the inside was with blocked fibers, that is, dead-ended under pressure in excess of the bubble point, or by the gas-distension method referred to herebelow, also under pressure in excess of the bubble point.

The second method for cleaning porous, elastic, hollow fibers from the inside, is the popular gas-distension method. This method comprises introducing a gas into the fibers under sufficient pressure to pass through the walls of the fibers, in a direction opposite to that in which the feed is being filtered, so as to dislodge solids retained on the walls of the fibers. This method is the subject of U.S. Pat. Nos. 4,767,539 and 4,921,610 to Ford, and related patents assigned to Memtec Limited. According to the '539 and '610 processes, for "outside-in" flow, gas is introduced into the lumens of the fiber as the back-wash medium, optionally after "back-flushing" ("back-washing" and "rinsing" are two other terms used interchangeably in the art with back-flushing) with permeate. Preferably the gas pressure in the lumens swells fouled fibers to enlarge their pores making it easier to free the particles lodged in the pores, and to carry them away in the expansion of the back-wash gas. Such a system is commercially available as a Memcor microfiltration system (Memtec).

To use the gas-back-flushing system effectively it is desirable to have highly elastic membrane walls which have pores which return to their original size after "explosive decompression" of gas through them. In such instances, one may first use a permeate back-flush and follow it with a gas back-flush. The chief drawback of the intermittent gas-pressurization process is that it places great strain on the membrane and relies on mechanically dislodging fouling matter which, for the most part is adhesively bonded to the membrane wall with physico-chemical forces such as Van der Waal's forces and the like, and perhaps also with covalent bonds.

As will be seen from the data presented in FIG. 5, back-flushing a polysulfone fiber at 175 kPa with permeate, or even deionized RO water, is far less effective than diffusion-cleaning with an oxidative anion such as a halogen, e.g. fluorine, chlorine, bromine or iodine. To obtain the desired explosive decompression of gas through the pores, the permeate side of the membranes is shut off, or "dead-ended".

Another, and older, method of cleaning fouled hollow tubes in particular, from the inside without draining the feed, requires back-flushing with permeate under relatively low pressure, particularly limited by the tolerance of the membrane to hydraulic pressure. The phrase "relatively low pressure" refers to pressure exerted by the gas-cleaning system which uses sufficient pressure to distend the membrane and dislodge foulant particles trapped in the membrane pores. As one would expect however, because back-flushing relies on loosening solid particles on the surface by forcing them off with hydraulic forces, it is not as effective as short bursts of pressurized gas. The hydraulic forces act over a much longer period of time than do the forces of a pressurized gas, and the time during which they act provides enough time for the hydraulic fluid to find a path of less resistance than that of the path blocked by fouling solids.

The hydraulic back-flushing system is also referred to as "dead-end" washing because the discharge of the manifold carrying fluid from of the bores of the fibers is blocked to allow the build-up of necessary hydraulic pressure above 240 kPa. The cleaning solution is held for a period of time under pressure, then drained through the discharge into a spent cleaning-solution tank.

This prior art back-flushing method is only effective when the cleaning solution is relatively non-toxic because a large portion of the cleaning agent escapes through pores which are not plugged, or only partially

plugged, and also through pores after they are cleaned and before the hydraulic pressure is removed. Since, after cleaning fibers in raw or "dirty" water, by back-flushing with toxic cleaning solution, clean water is withdrawn into the fibers as permeate, the toxic cleaning solution re-enters the fibers with the permeate. Even if the amount of cleaning agent re-entering with permeate is insignificantly small, a far greater amount of cleaning agent is used than is necessary to effect desirable cleaning. Finally, in the special instance where the fibers are withdrawing water from a medium containing live biomass, particularly a biomass which desirably helps purify the water, the discharge of a relatively large amount of toxic cleaning solution into the biomass kills so many cells that it takes an abnormally long period to return the biomass to its desired cell concentration, if it can be returned at all.

Further, to cope with the release of excess cleaning agent into the water to be purified, the cleaning agent is used infrequently, compensated by frequent back-flushing with permeate. Whether by forward or reverse flow, permeate helps significantly to maintain clean membrane surfaces. But back-flushing with permeate recycles it at the expense of permeate production and can only be justified when the cleaning effect of back-flushing is great enough to overcome the economic disadvantage. Thus substituting cleaning agent for gas in the '539 and '610 processes fails to provide a controllable, diffusion-controlled, substantially pressureless cleaning system.

Moreover, back-flushing a membrane's outer surfaces with biocidal solution, then back-flushing inner surfaces with permeate, is generally limited to processes in which the operating transmembrane pressure is relatively low, in the range from 1–3 bar, at which low pressure the solids are not forced into the pores of the membrane. In those instances when the flux is relatively low, in the range from 5 to 20 LMH, the fluid velocity of cleaning fluid to clean from the outside is too low. If cleaned with high velocity fluid the cleaning liquid enters the lumens, making this an unrealistic alternative.

It will now be appreciated that the cleaning systems which can be operated effectively without draining the feed, include those using pressurized back-flushing with a biocidal solution, such as in the Japanese system of JP 4-265127A and those using pressurized back-flushing with a gas, such as in the Ford '539 or '610 gas-distension systems.

It is not practical to back-flush fibers with permeate because the cleaning effect of permeate is solely due to hydraulic pressure and is therefore relatively ineffective. Further, to obtain a minimum liquid velocity of 1 meter/see of permeate through a lumen 1 mm in diameter, at a pressure below the bubble-point of the membrane, the pressure drop through the lumen is so high that a length of fiber only 1 meter, requires fiber-bursting pressure at the inlet to generate a pressure below the bubble-point, at some point downstream of the inlet. When the pressure does not exceed that which can be tolerated by the fibers, tubes or rolls, and they are back-flushed with permeate at such pressure, permeate is lost to the feed.

In the other methods, if the fibers are to be cleaned from the outside, the feed is shut off and drained, as is the permeate, the fibers are soaked in cleaning solution, washed and rinsed, on their outside surfaces, then finish-rinsed with fresh permeate before the membranes are returned to service.

Specifically with respect to hollow fiber membranes having an inside (lumen) diameter in the range from 0.5 mm to 5 mm, the feed is always on the outside. The i.d. of a fiber is at least 20 μm and may be as large as about 3 mm, typically being in the range from about 0.1 mm to 2 min. The larger the o.d., the less desirable the ratio of surface area per unit volume of fiber, but the lower the pressure drop for a back-flushing cleaning fluid. The wall thickness of a fiber is at least 5 μm and may be as much as 1.2 mm, typically being in the range from about 15% to about 60% of the o.d. of the fiber, most preferably from 0.5 mm to 1.2 mm.

The average pore cross sectional diameter in a fiber may vary widely, being in the range from about 5Å to 10,000Å. The preferred pore diameter for ultrafiltration of components in a substrate feedstream being in the range from about 5Å to 1,000Å; and for microfiltration, in the range from 1,000Å to 10,000Å.

It will now quickly be evident that a module containing fibers, whether held in arrays framed in wafers or frames, or held in oppositely disposed manifold means or "headers" in frameless arrays, may be viewed as being analogous to a liquid-liquid shell-and-tube heat exchanger. To clean fouled tubes in the exchanger is only possible in the unique situation where a first liquid is recycled through the tubes either to heat (or cool) a second liquid in the shell side, and the tube side gets frequently fouled. In this situation one may switch from recycling the first liquid to recycling a cleaning solution which can provide substantially the same heating (or cooling) function as the first liquid. After an appropriate amount of time, when the fouled tubes are clean enough, the cleaning solution is run into a cleaning solution holding tank and the first liquid is substituted.

Moreover, if one were to consider it, in the same manner as one might consider flowing cleaning solution through large diameter membrane tubes, the logical approach would be to pressurize the fibers with the cleaning solution from within, to reap the benefits of both (a) a higher flux for the cleaning solution, and (b) enlargement of the pores such as is obtained with the gas pressurization process. The obvious way to pressurize the fibers is to "dead-end" them, that is, to block the discharge of the cleaning solution from the outflow end of the lumens so as to force the cleaning solution out of the pores under high pressure greater than the bubble point of the membranes.

Assuming the membrane's performance is unaffected by an arbitrarily large number of dead-end back-flushing cycles, the problem with such cleaning is that it uses far more cleaning solution than is necessary, and is time-consuming compared to our cleaning method. Apart from the expense, since cleaning solutions are far from inexpensive, they are also highly toxic to bacteria which one may deliberately wish to keep in a biological treatment system containing plural frameless arrays, for their ability to biodegrade contaminants which may be present in the water.

An obvious drawback of cleaning from the outside of a tube or fiber, rather than from the inside, is that to do so requires a shell. If there is no shell, as in a frameless array such as one disclosed in the '524 array must be removed from the process reservoir in which it operates and immersed in a cleaning solution in another tank. An alternative is to drain the process reservoir and to substitute cleaning solution; then drain the cleaning solution after cleaning, and refill the reservoir. As is evident, this is a highly undesirable alternative.

Further, cleaning from the outside of a tube or fiber requires a large volume of cleaning solution since the system holdup volume must be filled. The permeate side volume is very small in comparison. Finally, any cleaning solution applied to the outer surface of a tube or fiber from the outside, is typically done under sufficient pressure to force the solution from outside the membrane through the biofilm on it and its pores. To save on time in the cleaning cycle, a relatively high pressure is applied, higher than is otherwise necessary, and such pressure has the effect of compacting the gel layer and foulants on the membrane wall, thus exacerbating the cleaning problem. Cleaning from the inside, particularly with continuous recirculation through the fibers, avoids using a higher pressure than is necessary to permeate the membrane wall, that is, a pressure no higher than that required to produce laminar flow on the membrane's permeate side, until its surface is sufficiently clean as evidenced by the restoration of a desirable flux.

SUMMARY OF THE INVENTION

Highly effective cleaning of a module containing an UF or a MF membrane having a fouled surface is obtained during an unexpectedly short period, without draining feed (substrate) from the module, by introducing a chosen cleaning fluid into the permeate and recycling it through the lumens at low pressure in the range from about atmospheric but no more than the bubble-point of the fiber. The method comprises maintaining a selected low pressure no more than the bubble-point either continuously, or cyclically applied, over a short period of time, preferably less than 1 hr, sufficient to diffuse enough cleaning fluid through pores in the membrane into the dirty water, substantially to re-establish the initial stable flux. The low pressure may be substantially constant, or it may be deliberately varied within a period of less than 5 sec, preferably less than 1 sec. When pulsed to achieve pulsed diffusion, the pressure exerted by the cleaning fluid may vary from a minimum of about 100 kPa (1 bar, at least 0.1 psig, preferably 0.5 psig) for a "loose" MF (5 μm) to a maximum of 100 psig for a "tight" UF (50Å), within less than 1 sec, which pulsing affords diffusion-controlled permeation. The pulsed maximum pressure which provides diffusion-controlled flow depends upon the pore size and distribution of the membrane but is generally no higher than about 300 kPa. Such flow discharges a predetermined amount of cleaning fluid into the feed and effectively removes the fouling film sufficiently to restore the transmembrane flux to within 20% of its initial stable flux over a period of 24 hr. The amount of cleaning fluid discharged into the feed is so small with each cleaning cycle that, even after an arbitrarily large number of cycles greater than 1000, continued withdrawal of permeate from the feed contaminated with cleaning fluid, does not deleteriously affect the permeate quality. In all cases diffusion through the wall of the membrane under diffusion-controlled flow occurs in a surprisingly short time, which provides for a short cleaning period; and a short cleaning period is a critical factor in the commercial attractiveness of a membrane separation.

The clean-in-place process of this invention does not dead-end the fibers to be cleaned, and it does not use high pressure; nor does the instant process physically dislodge fouled particles from pores in which they may be trapped with mechanical force or hydraulic force, but by chemical attack which affects the chemical bond between the fouling compound and the wall of the membrane. By so doing, the process capitalizes on the superior effectiveness of chemically removing a "foulant" (fouling material) whether organic or inorganic, in contrast with mechanically doing so by reliance on enough mechanical or hydraulic pressure to obtain measurable, or evident membrane wall distension known to loosen the mechanical bond of the foulant to the membrane's wall.

Specifically, an aqueous cleaning fluid comprising a biocidal oxidative electrolyte in aqueous solution, having an active, preferably oxidizing anion and an associated, preferably active cation, is found to migrate through partially blocked pores in a membrane and chemically attack organic and inorganic fouling matter on the surface of the membrane until the fouling matter is removed from the pores. The oxidizing anion may be contributed by an aqueous organic acid, particularly mono and polycarboxylic acids such as citric or oxalic acid and inorganic acids such as phosphoric acid. Alternatively, the cleaning fluid may be a gas which can diffuse through the pores of the membrane and chemically react with the foulant to remove it. Such gases may be biocidal, or oxidative, or both, and include sulfur dioxide, chlorine, fluorine, ethylene oxide and the like.

It is therefore a general object of this invention to provide a method for restoring the flux of a surface of a microfiltration or ultrafiltration semipermeable membrane after the surface is contacted with a non-sterile aqueous substrate such as dirty water containing inorganic material which can be deposited on the surface, or beneficial bacteria, from which substrate purified water is to be withdrawn. When the substrate includes the bacteria, the purified water is to be withdrawn without vitiating the benefits of the bacteria population. Whether the dirty water contains undesirable inorganic salts, particularly water-soluble halides, oxides and sulfides of the transition elements of Groups VI, VII and VIII of the Periodic Table, or organic matter, the dirty water being non-sterile usually contains enough bacteria to produce an initial biofilm on the surface of the membrane, which initial biofilm, with time, gets progressively denser or thicker, or both. Operation with the initial biofilm is unavoidable, but the membrane's initial stable transmembrane flux soon decreases as a function of time by at least 20%. The method of restoring the flux comprises, contacting the surface with a cleaning fluid at a pressure no higher than its bubble pressure breakthrough, but enough to diffuse through said pores and said film, over a period sufficient to remove enough fouling film to provide a restored flux equal to at least 70% of said initial stable flux; discontinuing contacting the surface of said membrane with the cleaning fluid; and, re-establishing flow of purified water through the membrane.

In the specific instance when cleaning hollow fiber membranes in a bioreactor containing a biomass, using a biocidal solution which is also an oxidative electrolyte having an oxidizing anion and an associated cation, the pressure is no greater than the bubble-point but sufficient to diffuse through the pores and the biofilm, but insufficient to kill numerically more than 20% of living bacteria in the biomass so as to maintain the viability of the bacteria population in the bioreactor; withdrawing the electrolyte from within lumens of the fibers; and, reestablishing normal operation. Most preferably, this is done without blocking the flow of the biocidal solution, but if desired, the flow of the solution may be blocked

so long as the pressure on the solution does not exceed the bubblepoint of the fibers, and the solution may be held in the lumens for long enough to remove most of the biofilm.

BRIEF DESCRIPTION OF THE DRAWINGS

The foregoing and additional objects and advantages of the invention will best be understood by reference to the following detailed description, accompanied by schematic illustrations of preferred embodiments of the invention, in which illustrations like reference numerals refer to like elements, and in which:

FIG. 1 is a bar graph depicting the results of a factorial analysis showing the average main effects and interaction of variables: time during which the cleaning fluid was in contact with the membrane, or "duration" (D), the concentration of the cleaning fluid (C), and the pressure of the cleaning fluid (P).

FIG. 2 is a perspective view schematically illustrating a membrane device disclosed in the '424 patent, comprising a frameless array of a skein of fibers, unsupported during operation of the device, with each set of the opposed ends of the fibers potted in one of two spaced apart headers, each atop and in open fluid communication with a permeate collection pan, and a permeate withdrawal conduit. By "unsupported" is meant "not supported during operation of the membrane device, except by the substrate".

FIG. 3 diagrammatically illustrates the cleaning of a cartridge of wafers comprising arrays of hollow fiber MF membranes, the cartridge being housed in a shell through which feed is flowed in outside-in flow.

FIG. 4 is a graph in which the variation of flux is plotted as a function of time, comparing the results obtained by back-flushing the lumens of polysulfone fibers with (i) permeate, (ii) deionized water, and (iii) a dilute solution of sodium hypochlorite (NaOCl) at concentrations which provided 150 ppm or 300 ppm "active" oxidizing anion all back-washed for the same amount of time, 30 min at a maximum continuous pressure of 245 kPa (30 psig).

FIG. 5 is a graph in which the variation of flux is plotted as a function of time, showing the results obtained by back-flushing for only 15 min per 24 hr of operation, the lumens of polyfluorovinylpyrrolidone fibers used to filter domestic wastewater having a high BOD_5 of 1,800 mg/L after the fibers are fouled sufficiently to halve their initial transmembrane flux of about 78 LMH.

FIG. 6 is a graph in which the variation of flux is plotted as a function of time, showing the results obtained by back-flushing for only 15 min per 24 hr of operation, the lumens of polyfluorovinylpyrrolidone fibers used to filter groundwater containing a high level 0.4 ppm of iron and manganese (2.1 ppm) after the fibers are fouled sufficiently to decrease their initial transmembrane flux by about 15%.

FIG. 7 schematically illustrates a single bank of 3 modules, in a large tank (not shown) of non-sterile ground water, each of which modules is similar in construction to the one with the frameless array shown in FIG. 1; and, the simplicity of the piping scheme to clean the bank in place, without having to drain the feed tank.

FIG. 8 diagrammatically illustrates the use in a single large body of bacterially contaminated water, such as a lake (not shown) of 4 banks, each having 3 modules, each of which banks is similar in configuration to the one shown in FIG. 7; and the simplicity of the piping scheme to clean all 4 banks in place, concurrently, without having to drain the feed tank.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

Referring to FIG. 1 there is shown a bar graph in which the results of a factorial analysis of data derived from in situ cleaning of polysulfone membranes were plotted. As is evident from the contribution of each variable to flux, plotted along the vertical axis, the duration of contact with the cleaning fluid is the variable with the most dominant effect. The next most dominant variable is concentration, followed by pressure which has the least effect. Since duration and pressure are the most influential variables, and these variables define the type of flow, we believe this flow to be diffusion-controlled flow.

The in situ cleaning process may be used in any membrane filtration system using hollow fiber membranes. The process is most particularly directed to water purification membranes such as are used in wastewater containing domestic sewage, chemicals, oily water, and pulp and paper byproducts; and, in surface water purification where the feed is brackish water or polluted lake water. In all of such environments the fouling film is to be removed sufficiently to restore the flux to desirable level relative to the initial stable flux. The in situ cleaning process is most preferred in particular situations where it is practical deliberately to kill no more than 20%, preferably $<10\%$ (cell count, CFU/ml) of the bacterial population in the interest of maintaining the beneficial effects of that population.

In all cases this invention relies on cleaning from the permeate side, that is, through the lumens of the fibers. In this manner, cleaning solution permeates through pores in the membrane and first reaches foulants embedded in those pores while the cleaning fluid is at its highest concentration, then permeates to the surface. The fluid thus has maximum effect on the foulant in the pore and in the fouling film.

Though less desirable than a liquid cleaning fluid, gaseous cleaning fluids such as chlorine, sulfur dioxide, ethylene oxide and the like are highly effective.

When a biocidal solution is used, it must first permeate the macroporous wall of the membrane in which essentially no bacteria are lodged (they cannot come through the skin and intermediate transport layers of a membrane) and attack bacteria, dead and alive, randomly lodged in the biofilm to provide a random network of pores through as much of the biofilm as is left. In general, there always is some biofilm left because the time over which diffusion takes place is insufficient to remove all the biofilm even if all the bacteria are killed in the biofilm.

The use of a biocidal solution which is incapable of diffusing through the biofilm easily will require too long a soak period and/or too long a recirculation period. Therefore the choice of biocidal solution is typically an oxidative electrolyte, and the concentration in which it is to be used, must be related to the transmembrane flux of that solution through the membrane to be cleaned and to the foulant(s) to be removed. By "oxidative electrolyte" we refer to one which at least has an active anion, and preferably also an active associated cation and include such materials as the organic peroxides and hydrogen peroxide. Preferred biocidal solutions and the foulants for which they are generally particularly effective are listed side-by-side in Table herebelow:

TABLE

Cleaning solution	Foulant
Hydrochloric acid, HCl – pH 4	Inorganic solids, $CaCO_3$
2.0 wt % citric acid + NH_4OH – pH 4	Inorganic colloids, metal oxides, $CaCO_3$
NaOH – pH 11	Organics, inorganic colloids, silica
0.25 wt % HCHO followed by a detergent (with phosphate)	Biological matter
NaOCl with 100 ppm "active" Cl – pH 5 to 10	Organics, biological matter
1 wt % NaCl	General cleaning
1 wt % oxalic acid – pH 2 to 4	Colloids, iron oxides
1 wt % $NaHSO_3$ – pH 5 to 6	Colloids, iron oxides
700 ppm EDTA/2500 ppm NaEDTA – ph 6	Metals, $CaCO_3$, $MgCO_3$; oxide or sulphate scales

It will be noted that the term "solution" is used since it is most convenient to use an aqueous biocidal solution of known concentration. If desired, non-aqueous liquid oxidant may be used if the amount diffusing through the membranes can be controlled. For example, fuming nitric acid, chloracetic acid, or non-aqueous HCl may be injected into permeate held in the piping and lumens, but it is difficult to inject just the right amount. Besides being a difficult "handling" problem, non-aqueous cleaning fluids are difficult to meter accurately in the minuscule amounts required.

The cleaning fluid chosen is preferably inert relative to the synthetic resinous material of the membrane though it may swell in contact with the cleaning fluid; for example, polypropylene fibers tend to be hydrolyzed with NaOCl solution, but are inert with respect to aqueous H_2O_2 (hydrogen peroxide); and, polysulfone fibers tend to swell in contact with NaOCl solution but are otherwise inert to the solution. Depending upon the toxicity to the bacteria population, as little as 10 ppm of the cleaning fluid can be effective.

With particular reference to a cleaning fluid which is a conventional biocidal oxidative electrolyte, a concentration no greater than 500 ppm of the active anion, e.g. OCl^-, or Cl^- is preferred, since higher concentrations up to 0.1% by weight of the active anion fails to provide significantly improved performance. The temperature of the biocidal solution as well as its concentration may be raised provided neither is deleterious to the membrane, and th increased concentration provides a justifiable effectiveness of "kill" without jeopardizing the vitality of the bacteria population.

The fibers used in an array may be formed of any conventional organic membrane material. They are typically polymers which form an asymmetric membrane having a thin layer or "skin" on the outside or "shell side" of the fibers. Preferred materials for a base membrane which do not contain a repeating unit derived from acrylonitrile, are polysulfones, poly(styrenes), including styrene-containing copolymers such as butadiene-styrene and styrene-vinylbenzylhalide copolymers, polycarbonates, cellulosic polymers, polypropylene, poly(vinyl chloride), poly(ethylene terephthalate), poly(vinylidene fluoride), aromatic polyamides and the like disclosed in U.S. Pat. No. 4,230,463 the disclosure of which is incorporated by reference thereto as if fully set forth herein.

The fibers are chosen with a view to performing their desired function and are non-randomly oriented in each array, and in the module as described in the '424 patent, the disclosure of which is incorporated by reference thereto as if fully set forth herein. In a frameless array such as is shown in FIG. 2, the direction of the flow of feed is immaterial as the direction in which the feed enters a lumen is generally transverse to the upstanding fibers. In a module housing one or more cartridges of wafers such as are shown in the '593 patent to Pedersen et al, the flow of feed through the module is over the fibers and orthogonal thereto. It is preferred to use banks of modules constructed as disclosed in the '424 patent, the disclosure as to the construction of which is incorporated by reference thereto as if fully set forth herein.

Typical hollow fiber membranes which are particularly amenable to being cleaned in situ have an i.d. in the range from 0.5 mm to 2.5 mm and have an o.d. in the range from 0.7 mm to 3.5 mm.

The average pore cross sectional diameter in a fiber may vary widely, being in the range from about 5Å to 2000Å. The preferred pore diameter for separation of components in a liquid feedstream is in the range from about 10Å to 200Å.

Unlike in a conventional module, the length of a fiber in a skein is essentially independent of the strength of the fiber, or its diameter, because the skein is buoyed, both by bubbles of oxygen-containing gas introduced if live aerobic bacteria are present, and the substrate in which it is deployed. The length of each fiber in the skein is preferably determined by the conditions under which the array is to operate. Typically fibers of a skein range from 1 m to about 10 m long, depending upon dimensions of the body of substrate (depth and width) in which the array is deployed. For the longer fiber, a larger diameter membrane is desirable to minimize the pressure drop through the fiber.

The number of fibers in an array is arbitrary, typically being in the range from about 1,000 to about 10,000, and the preferred surface area for a skein in commercial service is in the range from 10 m^2 to 100 m^2.

The materials for the headers are most preferably either thermosetting or thermoplastic synthetic resinous materials, optionally reinforced with glass fibers, boron or graphite fibers and the like. Thermoplastic materials are preferred for relatively low temperature service below 100° C., these being chosen so as to be sufficiently compatible with the material of the fibers to produce a lasting, fluid-tight bond. Such thermoplastic materials may be crystalline, such as polyolefins, polyamides (nylon), polycarbonates and the like, semi-crystalline such as polyetherether ketone (PEEK), or substantially amorphous, such as poly(vinyl chloride) (PVC), and the like. Thermosetting resins are preferred for higher temperature service, and for ease of use.

The particular method of securing the fibers in each of the headers is not narrowly critical, the choice depending upon the materials of the header and the fiber, and the cost of using a method other than potting. However, it is essential that each of the fibers be secured in fluid-tight relationship within each header. This may be effected by simply not bundling the terminal portions of the fibers too tightly before potting them.

Since there is very little hydraulic pressure, typically less than 1.33 bar (5 psig) exerted by the cleaning fluid in the pores of the membrane while the fluid is recirculated through the membrane, and insufficient pressure to cause hydraulic flow of solution through the pores even if pulsed, the flux obtained with the solution, is essentially diffusion-controlled and foulants lodged in the pores cannot be dislodged by hydraulic pressure. Instead, foulants are dissolved or degraded by chemical

action. The main purpose of pulsing is to avoid, to the extent possible, diffusion flow through pores which are already open and offer the path of least resistance. Pulsing at low pressure, less than about 20 psig (240 kPa) tends to distribute the biocidal solution randomly and isotropically under the inner surface of the membrane.

Reverting to FIG. **2** there is shown in perspective view a membrane device referred to generally by reference numeral **10**, comprising an upstream header **11** and a downstream header **11'**, one being substantially identical to the other, upstream and downstream collection pans **15** and **15'** to collect the permeate, and their respective permeate withdrawal conduits **17** and **17'**. The purpose of the headers **11** and **11'** is to pot fibers **12** in spaced apart relationship with each other in a potting resin such as an epoxy. The headers are conveniently formed as described in the '424 patent, but any other method may be used which serves the aforementioned purpose. The bases **13** and **13'** of each header are snugly accommodated in collection pans **15** and **15'** sized to the base **13** above a permeate collection zone within the pan. Air is provided through a gas distribution means **19** to maintain beneficial bacteria present in the dirty water. Permeate withdrawn into the lumens of the fibers, preferably under suction, collects in the pans and is discharged to a collection point as is described in the '424 patent, until the flow of permeate is about one-half of the flow at initial stable flux, at which time the flow of dirty water is shut off so that the lumens of the fibers remain filled with permeate, and the cleaning cycle is commenced.

Conduits **21**, **22** and **23** are provided as shown, connecting the lumens of fibers **12** in valved communication with the discharge of a pump **24** via a 3-way valve **25**, which in one of its positions allows permeate to be withdrawn from the headers. Conduit **22** serves as a manifold for the collection pans **15**, and an intermediate portion **22'** of the conduit **22** is provided with a check valve **26** which allows biocidal solution held in cleaning tank **27** to be circulated through the lumens of fibers **12**, and returned through conduit **23** to the tank **27**. A check valve **28** is provided in conduit **23** to shut off flow of either permeate or biocidal solution to the cleaning tank.

The 3-way valve **25** is positioned to flow biocidal solution to the upstream collection pan and enough solution is pumped from tank **27** to fill the upstream collection pan and the lumens of the fibers **12**, then flow into the downstream collection pan from which it is returned to the tank **27**. Check valve **23** is left open when cleaning solution is either circulated with pump **24** or pulsed when a pulse pump is substituted for pump **24**. In those instances where it is desired to "dead end" the biocidal solution under only enough pressure to permit its diffusion-controlled flow out of the fibers, both the check valves **26** and **28** are closed.

Referring to FIG. **3**, there is shown a module **40** having a shell **41** within which at least one cartridge **42** of wafers (only the rectangular-mesh protective screen **43** on the topmost wafer is visible) is disposed between upper and lower feed plates **44** and **44'** (not visible in this view) which are longitudinally axially connected with diametrical baffles **45** and **45'** which extend the length of the shell and fit in fluid-tight relationship with diagonally opposed ends **46** and **46'** of the cartridge so that the permeate side of the shell is divided into two separate permeate withdrawal zones. The fibers in each wafer are in parallel spaced apart relationship and discharge permeate under suction conditions into both permeate withdrawal zones when dirty water is flowed axially through the center of the module as described in greater detail in the '593 patent.

Again, when the flow of permeate is about one-half the flow at initial stable flux, indicating the flux has decreased to about half, the feed is shut off and the cleaning cycle commenced. The feed does not need to be shut off since it does not interfere with the effectiveness of the cleaning cycle. However, the bubblepoint may change depending upon the exerted hydrostatic pressure.

As illustrated in FIG. **3**, biocidal solution is circulated through conduits analogous to those used in the prior embodiment, except that a 3-way valve **29** is substituted for check valves **26** and **28** in FIG. **2**. In the positions shown, the 3-way valves indicate that permeate is being withdrawn from the module **40** through permeate withdrawal conduits **17** and **17'**. As before when it is desired to clean the outer surfaces of the fibers, biocidal solution is circulated through their lumens until the flux is restored to at least 70% of the initial stable flux, and preferably to more than 80%. After the biocidal solution is drained to the tank **27**, permeate withdrawal in normal operation is re-commenced. As before, the flow of dirty water need not be shut off. If shut off the dirty water remains in the casing outside the tube and in contact with the biofilm on the outer surface of the membrane **54**.

Referring to FIG. **4**, there is plotted the results of a pilot plant test in which the effect of various back-flushes, each having a duration of 30 min, and carried out sequentially, was evaluated. The integers in brackets identify the value of the flux after the array was back-flushed with the solution/water/permeate identified, as follows: (1) 300 ppm Cl as NaOCl solution at 170 kPa (10 psig); (2) RO water at 170 kPa; (3) RO water at 170 kPa, dead-ended; (4) permeate at 170 kPa; (5) 150 ppm Cl as NaOCl solution; (6) 300 ppm Cl as NaOCl solution at 150 kPa.

The foregoing tests were carried out with a frameless array of polysulfone fibers in a module analogous to one shown in FIG. **2**, comprising 110 MF fibers each 2 meters long, having an o.d. of 1.5 mm, an i.d. of 1.0 mm, and pores having a nominal diameter of about 0.15 μm, the majority of which are smaller than 0.15 μm, the smallest being about 0.08 μm and the largest 0.35 μm, as determined by liquid displacement porometry. The array is fully immersed in a tank deep enough to immerse the vertex of the parabolic array which vertex is about 0.75 meter above the bottom of the tank. Domestic wastewater is fed to the tank. As is evident from FIG. **5**, the initial flux is about 44 LMH, but the initial stable flux after a soak period of 4 hr is 38 LMH under a permeate withdrawal suction of 25.4 cm of Hg. After 72 hr the flux decreases to about 12 LMH, and the permeate being withdrawn is drained to storage. Without moving the array, the piping is configured to recycle a 300 ppm Cl NaOCl solution through the lumens by positioning the 3-way valve **25**, closing check valve **26** and opening check valve **28** (see FIG. **3**). On the scale illustrated, the 30 min period for back-flushing is not visible. Though restoration to the initial stable flux is not instantaneous (as evident from the inclination of the near-vertical line) after circulation of the biocidal solution is stopped, it is clear that the recovery is rapid.

The pressure of 170 kPa was arrived at by trial and error for the particular fibers used, this pressure being

sufficient to provide diffusion-controlled flow, the rate of which was not noticeably changed between 150–170 kPa. At 190 kPa the rate of flow was noticeably increased indicating flow under pressure due to developed hydraulic forces.

The 300 ppm OCl^- concentration was arrived at with a little trial and error during which it was determined that higher concentrations provided a rapidly increasing "kill" of cells in the medium without a correspondingly high improvement in flux; lower concentrations provided correspondingly lower kills and unnecessarily prolonged the time required to attain the initial stable flux.

The biocidal solution was made from a commercially available Javex bleach solution containing 5.25% NaOCl, and 300 ppm was made up according to the following calculations:

$$NaOCl \rightarrow Na^+ + OCl^- \quad Mw\ 74.5\ 51.5$$

so that 1.45 g NaOCl yields 1 g OCl^-

and for a 300 ppm OCl^- solution the concentration of Javex solution needed is (1.45 g NaOCl/g OCl^-)(1 ml Javex/0.0525 g NaOCl)(300 mg/L) =8.28 ml Javex solution/L of water

It is evident from the data presented in FIG. 4 that the initial cleaning with 300 ppm OCl^- restored the flux to (1), essentially its original value. During the next cycle of permeate withdrawal, flux measurements were made every 12 hr. As seen, the last two measurements were substantially identical at 24 LMH when the back-flushing cycle was initiated with RO water which restored the flux to (2), about 42 LMH. When the back-flushing was repeated with RO water at the same pressure as the previous cycle, except that the check valve **28** was closed so the RO water was dead-ended. This was expected to provide better cleaning than was obtained with RO water which was not deadended, but the flux was restored only to (3) about 36.5 LMH.

The following cleaning cycle was not started until the flux had deteriorated from 36.5 LMH to about 16.5 LMH, when the tank of cleaning solution was emptied, and the permeate diverted into it. The array was then back-flushed with permeate which was recirculated through the array for 30 min at 170 kPa. The flux was restored to (4), about 25 LMH.

To determine the effect of a half-strength biocidal solution, when the flux had decreased from 25 LMH to 18 LMH, the array was back-flushed with 150 ppm OCl^- solution for 30 min at 170 kPa. The effect was to restore the flux to a value of 33.5 LMH (5) which was higher than the flux (25 LMH) before it decreased.

The following cleaning cycle was initiated when the flux decreased from 33.5 LMH to 19 LMH, when the array was back-flushed with 300 ppm OCl^- solution for 30 min at 150 kPa, a lower pressure than was used in cycle (1). The effect was to restore the flux to 39 LMH which is substantially the same as the initial stable flux.

It is evident from the foregoing that the effectiveness of the biocidal solution even at the low pressure of 170 kPa and low concentration of 300 ppm OCl^- was excellent.

Referring to FIG. 5, there is plotted the results of a pilot plant test in which a frameless array analogous to that shown in FIG. 2, of 1400 MF fibers each 2 meters long, having an o.d. of 2 mm, an i.d. of 1.5 mm, and pores having a nominal diameter of about 0.15 μm, the majority of which are smaller than 0.15 μm, the smallest being about 0.08 μm and the largest 0.35 μm. The array is fully immersed in a tank into which domestic wastewater is fed. The initial stable flux after a soak period of 4 hr is 78 LMH. When, after 24 hr the flux decreases to about 46 LMH, the permeate in the lumens is drained to permeate storage, and the piping configured for circulating the 300 ppm OCl^- biocidal solution as described hereinabove for FIG. 4. As before, the 15 min period for back-flushing is not visible on the graph. Again, from the steep, nearly vertical rise of the flux recovery, it is evident that restoration of the flux was rapid.

Details of the run in FIG. 4 over a period of 10 days are as follows:

Influent flowrate	9.408	L/min
Influent suspended solids	1800	mg/L
Mixed liquor temperature	25°	C.
Mixed liquor suspended solids	15,800	mg/L
Mixed liquor volatile suspended solids	13,700	mg/L
Mixed liquor dissolved solids	1,300	mg/L
Mixed liquor BOD_5	600	mg/L
Mixed liquor COD	14,400	mg/L
Mixed liquor pH	7.2	
Membrane outer surface area	13	m^2
Operating suction on permeate side	25.4	cm Hg (35 kPa)
Airflow to module	15	SCFM
Pressure of biocidal solution	5	psig
Flowrate of biocidal solution	2	L/min
Volume of biocidal solution diffused into tank	2	L
Permeate turbidity	0.600	NTU
Permeate BOD_5	<1	mg/L
Permeate COD	35.9	mg/L
Suspended solids in permeate	<1	mg/L
Total coliform count in permeate	12	CFU/100 ml

Referring to FIG. 6, there is plotted the results of a pilot plant test for recovering purified water from groundwater flowing into a tank in which a frameless array analogous to that shown in FIG. 2, is immersed. As permeate is withdrawn, the groundwater is concentrated into an aqueous substrate. A portion of this substrate is purged either continuously or periodically, to maintain a desired concentration of contaminants in the substrate.

The array used 110 MF fluoropolymer fibers each 2 meters long, having an o.d. of 2 mm, an i.d. of 1.5 mm, and pores having a nominal diameter of about 0.15 μm, the majority of which are smaller than 0.15 μm, the smallest being about 0.08 μm and the largest 0.35 μm. The array is fully immersed in a tank into which the groundwater contaminated with iron and manganese salts, is fed. The initial stable flux after a soak period of 4 hr is 90 LMH. When, after 24 hr the flux decreases to about 73 LMH, the permeate in the lumens is drained to permeate storage, and the piping configured for circulating the citric acid @pH 2.5 as described hereinabove for FIG. 4. As before, the 15 min period for back-flushing is not visible on the graph. Again, from the steep, nearly vertical rise of the flux recovery, it is evident that restoration of the flux was rapid. After 5 permeate withdrawal and cleaning cycles, it is evident that there is no substantial loss of flux relative to the initial stable flux.

Details of the run with groundwater in FIG. 6 over a period of 120 hr are as follows:

Influent flowrate	1.0	L/min
Influent iron	0.4	ppm
Influent manganese	1.1	ppm
Substrate temperature	14°	C.

-continued

Concentration of iron in substrate	3.3	ppm
Concentration of manganese in substrate	2.1	ppm
pH of substrate	10.5	
Cleaning solution	citric acid	at pH 2.5
Duration - cleaning period	15	min/24 hr
Pressure of citric acid solution	5	psig
Circulation rate of citric acid	2	liters/min
Membrane surface area	1	m^2
Operating suction, permeate side	25.4	cm Hg (35 kPa)
Airflow to module	0.28	m^3/min (1.5 SCFM)
Permeate turbidity	0.600	NTU
Permeate iron	0.06	ppm
Permeate manganese	0.05	ppm

Referring to FIG. 7 is schematically illustrated the use of 3 modules of frameless arrays of fibers freely swaying in skeins above headers which are manifolded for withdrawal of permeate from the lumens, in the medium of a reservoir in which beneficial aerobic bacteria are nourished. Conduits for supplying air under the skeins are not shown. As indicated, the cleaning cycles of each module may be undertaken separately, or they may be cleaned together. In each case, the flow of cleaning solution is not blocked through the skeins of fibers.

Referring to FIG. 8 is schematically illustrated another, larger use than that described in FIG. 7. Again, in the medium of a reservoir in which beneficial aerobic bacteria are nourished, 4 banks of 3 modules each are manifolded for withdrawal of permeate from the lumens. As indicated, the cleaning cycles of each bank may be undertaken separately, or they may be cleaned together. In each case, the flow of cleaning solution is not blocked through the skeins of fibers.

Having thus provided a general discussion, described the overall cleaning process in detail and illustrated the invention with specific examples of the best mode of cleaning fiber membranes in a module containing the membranes, it will be evident that the invention has provided a simple but effective solution despite the teachings of the art. It is therefore to be understood that, no undue restrictions are to be imposed on the scope of this invention by reason of the specific embodiments illustrated and discussed, and, particularly that the invention is not to be restricted to a slavish adherence to the details set forth herein.

We claim:

1. In a system for withdrawing permeate from a multicomponent liquid substrate having particulate matter and a population of beneficial aerobic bacteria suspended therein, with a gas-scrubbed assembly comprising a frameless array of hollow fiber membranes in combination with a gas-distribution means, said system comprising, a reservoir containing a volume of at least 100 liters of said substrate from which a permeate is to be withdrawn; a pair of headers adapted to be mounted in spaced-apart relationship within said substrate without being confined in a modular shell, a first header having terminal end portions of a multiplicity of hollow fibers secured therein, and a second header having opposed terminal end portions of said hollow fibers secured therein, essentially all ends of said hollow fibers being open so as to discharge permeate through said headers, at least one header being disposed below a horizontal plane through the horizontal center plane of said one header; said hollow fibers formed from a material selected from the group consisting of an inorganic material and an organic synthetic resinous material, and swayably buoyantly deployed as a skein in a body of said substrate, said hollow fibers together having an outer surface area in excess of 10 m^2, each fiber having a length >0.5 m and sufficiently greater than the direct distance between said first and second headers, so as to present, when said skein is deployed, a generally arcuate configuration above a plane through the horizontal center-line of a headers; permeate collection means for collecting said permeate; means for mounting said spaced-apart headers in open fluid communication with said permeate collection means; means for withdrawing said permeate; and, said gas-distribution means disposed within a zone beneath said skein, and adapted to generate bubbles which flow upwardly through said skein, whereby said hollow fibers are kept awash in bubbles and resist the build-up of said particulate matter on the surfaces of said hollow fibers on which is generated a biofilm clogging pores of said membrane, yet affords an initial stable transmembrane flux which decreases during each withdrawal period as a function of time by at least 20%, the improvement comprising,

(i) a container containing an aqueous solution of a biocidal oxidative electrolyte;

(ii) a conduit having a pump means in fluid connection with said fibers to flow said electrolyte in laminar flow through the lumens of said hollow fibers to permeate through said outer surfaces and said biofilm, at a pressure no higher than said membrane's bubble breakthrough pressure, but enough pressure to diffuse said solution through said pores and said biofilm, for a period sufficient to oxidize organic matter within said pores and in said biofilm so as to form a random distribution of pores through said biofilm and provide a restored flux equal to at least 70% of said initial stable flux;

(iii) conduit means operatively placing said permeate collection means in selectively open flow communication with said container; and,

(iv) valve means operatively connected in said conduit means to alternately withdraw permeate from said collection means, and to recirculate said electrolyte through said hollow fibers.

2. The system of claim 1 wherein said bubble pressure breakthrough is no more than about 300 kPa (30 psig).

* * * * *

US006331251B1

(12) **United States Patent**
Del Vecchio et al.

(10) **Patent No.: US 6,331,251 B1**
(45) **Date of Patent: Dec. 18, 2001**

(54) **SYSTEM AND METHOD FOR WITHDRAWING PERMEATE THROUGH A FILTER AND FOR CLEANING THE FILTER IN SITU**

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(73) Assignee: **Envirogen, Inc.**, Lawrenceville, NJ (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **09/329,538**

(22) Filed: **Jun. 10, 1999**

(51) **Int. Cl.**[7] **B01D 65/02**

(52) **U.S. Cl.** **210/636**; 210/195.2; 210/257.2; 210/321.69; 210/500.23; 210/805

(58) **Field of Search** 210/139, 194, 210/195.2, 257.2, 321.69, 636, 805, 247, 500.23, 650

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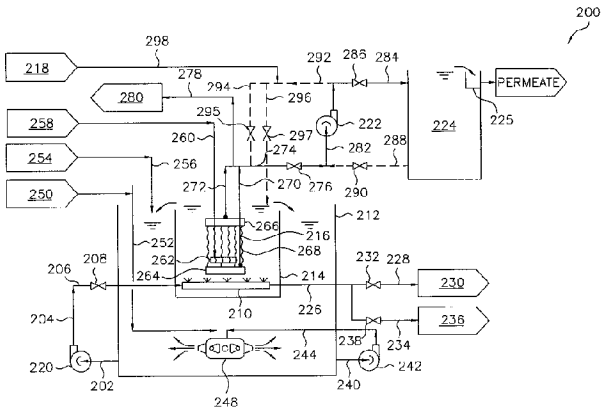
(74) *Attorney, Agent, or Firm*—Ratner & Prestia

(57) **ABSTRACT**

A system is provided for withdrawing permeate from a substrate through a filter and for cleaning the filter in situ. The system includes a vessel configured to contain a substrate and a compartment connected to receive substrate from the vessel and connected to return a portion of received substrate to the vessel during normal operation of the system. The system also includes a filter positioned at least partially within the compartment to withdraw permeate from substrate in the compartment during normal operation of the system. A source of cleaning solution is connected to the compartment to deliver cleaning solution into the compartment and into contact with the filter during cleaning operation of the system. The compartment facilitates circulation of substrate in the vessel during normal operation of the system and substantially prevents introduction of cleaning solution from the compartment into contact with substrate contained in the vessel during cleaning operation. A method is also provided.

26 Claims, 3 Drawing Sheets



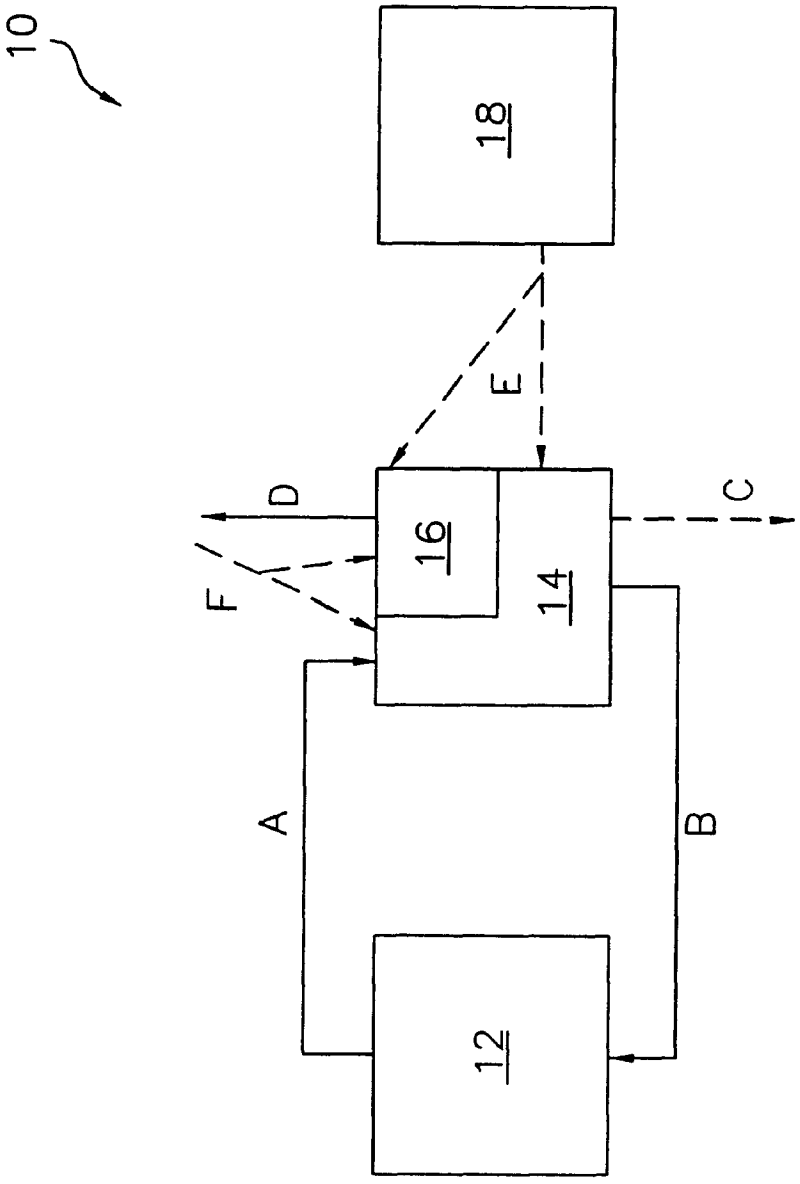


FIG. 1

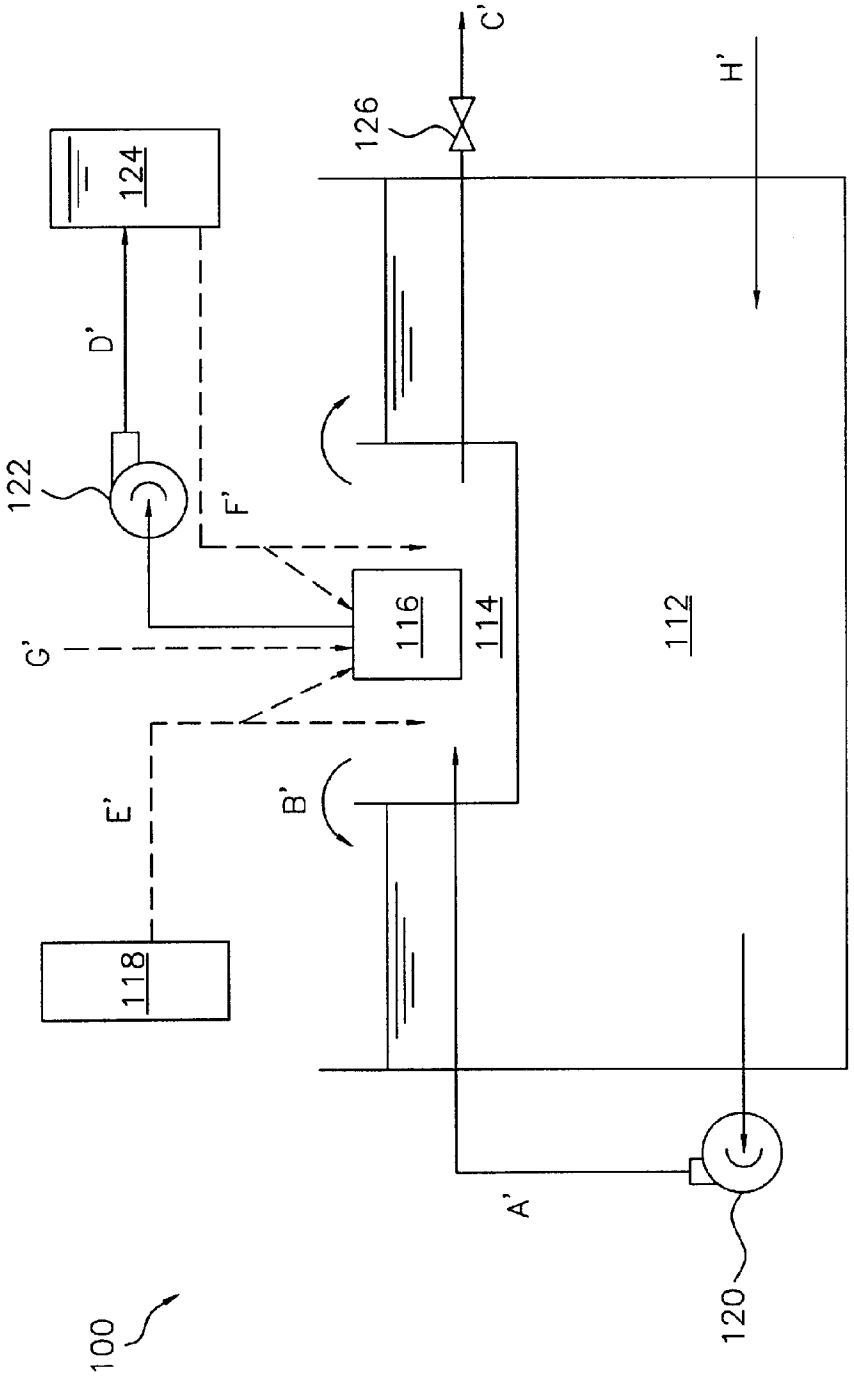


FIG. 2

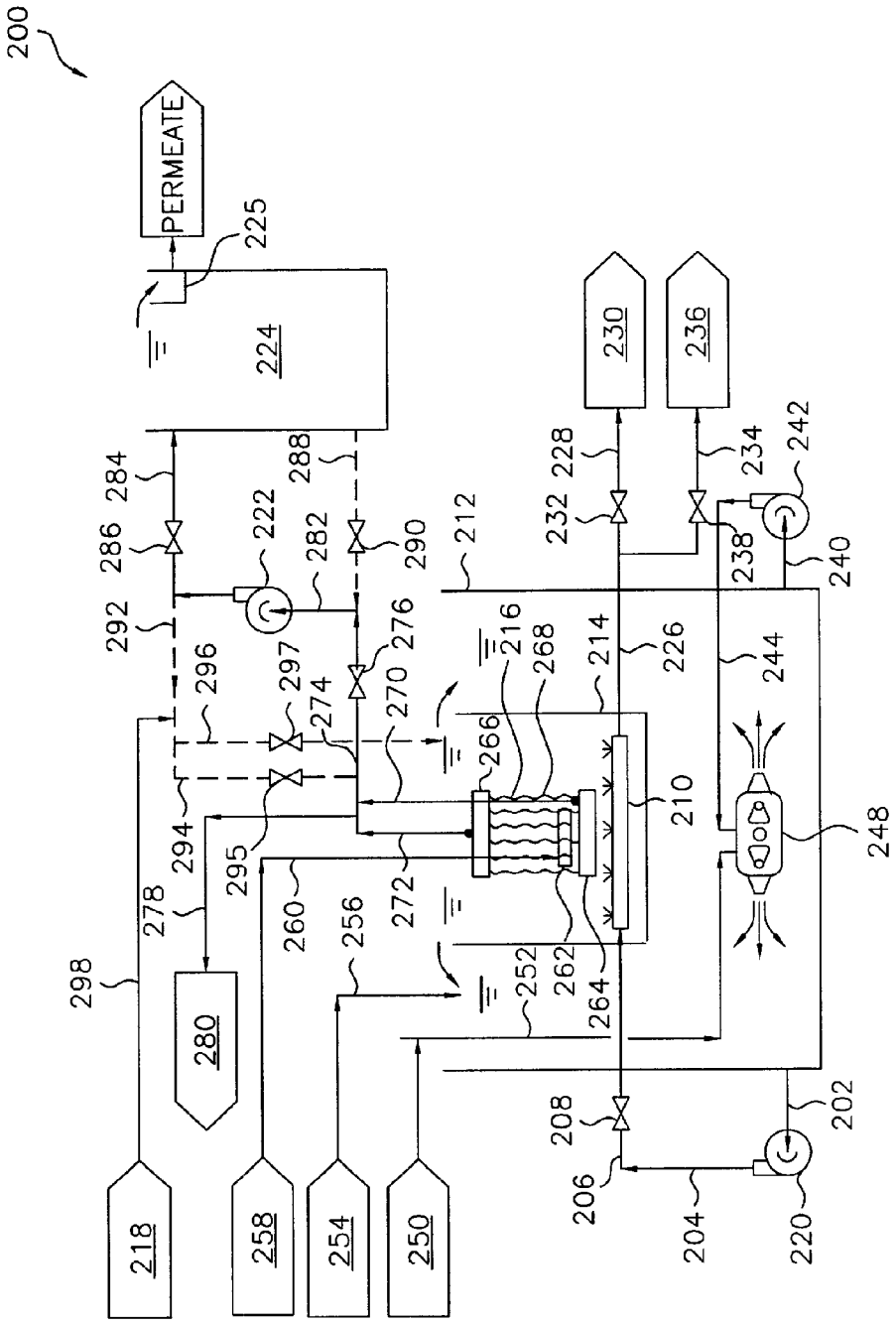

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FIG. 3

SYSTEM AND METHOD FOR WITHDRAWING PERMEATE THROUGH A FILTER AND FOR CLEANING THE FILTER IN SITU

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to a system for withdrawing permeate from a substrate through a filter. More particularly, this invention relates to a system adapted for withdrawing permeate from a substrate through a filter and for cleaning the filter in situ. A method is also provided.

2. Related Art

Filtration systems provide barriers in order to allow permeate to be drawn off from a substrate through the filter while concentrate is left behind. For example, filtration systems have been used as barriers to retain biosolids in biological reactors. In such filtration systems, membranes have been proposed as the barrier. Such membranes can be provided in the form of hollow fibers, tubes, or rolls, for example.

For the purpose of illustration, leachate treatment systems for wastewater treatment applications may use a membrane separator in order to separate feed into permeate and biomass. Such systems are available, for example, under the trademark ZEEWEED from Zenon Environmental Inc. of Ontario, Canada. The ZEEWEED system uses a submersible membrane cassette to bring about bio-oxidation to oxidize organic matter in the feed. Membranes are used to retain bacteria in the system for essentially complete oxidation and to provide high effluent quality.

It has been recognized that it is important to keep membranes used in such systems "clean" because, after some period of use, a fouling film or "bio-film" can form on the membrane, thereby reducing the flow of permeate through the membrane. A buildup, whether organic or inorganic, may form on the membrane's outer surface, inner surface, and/or in the membrane's pores that extend through the membrane's wall. Such a buildup on the membrane has, therefore, been recognized to decrease the performance of the membrane as a viable filter.

U.S. Pat. No. 5,403,479, issued to Smith et al. ("In Situ Cleaning System for Fouled Membranes") provides ample background as to the nature and extent of the fouling problem that tends to plague the bio-filtration industry. U.S. Pat. No. 5,403,479 is incorporated herein by reference in its entirety. As one possible solution to the problem of membrane fouling, Smith et al. proposed in the '479 patent a cleaning system for substantially restoring transmembrane flux in fouled, porous/semipermeable microfiltration or ultrafiltration membranes used to recover purified water from contaminated or "dirty" water. Specifically, Smith et al. proposed cleaning a module containing a membrane, without draining feed from the module, by introducing a chosen cleaning fluid into the permeate and recycling it through the lumens of hollow fiber membranes at low pressure not exceeding the bubble point of the fiber. The process proposed by Smith et al. in the '479 patent cleans from the permeate side of the membrane; that is, through the lumens of the hollow fibers.

U.S. Pat. No. 5,248,424, issued to Cote et al. ("Frameless Array of Hollow Fiber Membranes and Method of Maintaining Clean Fiber Surfaces While Filtering a Substrate to Withdraw a Permeate") proposed another approach for maintaining the performance of filtration membranes; more

specifically, a frameless array of hollow fibers. Cote et al. proposed in the '424 patent a system to reduce the build-up of growing microbes or the deposits of inanimate particles upon the surfaces of fibers kept awash in bubbles of a fiber-cleansing gas ("scrubbing gas"), particularly an oxygen-containing gas ("air-scrubbed"). The build-up is essentially naked when the fibers are buoyantly freely swayable in a frameless array submerged in a substrate through which the bubbles rise with sufficient physical force of impact to keep the fibers essentially free of deleterious deposits. Similar solutions were proposed by Mahendran et al. in U.S. Pat. No. 5,639,373 ("Vertical Skein of Hollow Fiber Membranes and Method of Maintaining Clean Fiber Surfaces While Filtering a Substrate to Withdraw a Permeate") and by Henshaw et al. in U.S. Pat. No. 5,783,083 ("Vertical Cylindrical Skein of Hollow Fiber Membranes and Method of Maintaining Clean Fiber Surfaces").

In International Publication No. WO 98/37950 ("Portable Reverse Osmosis Unit for Producing Drinking Water"), Daly et al. proposed a method and apparatus for producing drinking water from impure water wherein hollow tubular membranes of the system are periodically back flushed with retentate by directing the retentate to the inside surfaces of the membranes and by passing the retentate through the membranes, thereby dislodging particles from the outside surfaces. When chemical cleaning of the membranes is required in the method and apparatus proposed in the '950 publication, cleaning solution is pumped from a tank into the membranes.

In Australian Patent Application No. AU 9676300 (corresponding to International Publication No. WO 97/18887), Cote et al. described a method for cleaning immersed membranes in situ, wherein effluent contained in the tank is at least partially emptied in order to expose the membranes to the air, and cleaning solutions are passed through the pores of the membranes along a flow path opposite to the filtration flow of the effluent by delivering cleaning solution to the permeate side of the membranes. A shut-off valve is opened in order to drain off effluent from a treatment tank. Cleaning solution is then introduced into the membranes from a reservoir. In another embodiment, four tanks are supplied with effluent. When one wishes to clean the membranes in one of the tanks, the contents of the selected tank are transferred into the other tanks. Cleaning solutions are fed into the membranes of the empty, selected tank from reservoirs.

Although significant effort has been expended to resolve this recognized problem of fouling, improvements regarding the "cleaning" of filtration systems such as those that employ membranes are still in demand, whether the membranes are provided in the form of hollow fibers, tubes, rolls, or other membrane configurations. Specifically, despite these significant advances in the art of filter cleaning, and despite the purported ability of such proposed systems to prolong the throughput rate of the membranes used as filters, it has been discovered that, in some instances, the membranes must eventually be removed from the process for a thorough cleaning such as a deep chemical cleaning. The need to remove a filter from a system such as a biological reactor is of course time consuming, expensive, labor intensive, and generally undesirable. Moreover, it often requires that the system be at least partially shut down during the cleaning process while the filter is removed.

For example, it is undesirable to remove a submersible membrane unit from a biological reactor and to move the submersible membrane unit to a separate tank for cleaning. Membrane assemblies can be quite large and quite heavy.

Also, in the case of an industrial biological reaction system, the biological reactor vessels in which membrane assemblies are used can be quite tall, thereby requiring expensive and cumbersome rigging equipment for removal. Furthermore, the various "plumbing" connections to such membrane assemblies must be disconnected and subsequently reconnected in order to bring about membrane assembly removal and replacement, respectively. It will also be understood that over-head clearance may not be available for removing such membrane assemblies easily, and when such systems are removed, the process of doing so can create quite a mess. Also, external tanks dedicated to separate cleaning operations for off-line cleaning procedures require significant floor or ground space and numerous "plumbing" connections.

Accordingly, the need remains for an improved system for withdrawing permeate from a substrate through a filter and for cleaning the filter in situ. A corresponding method is also needed.

SUMMARY OF THE INVENTION

A system is provided according to this invention for withdrawing permeate from a substrate through a filter and for cleaning the filter in situ. The system includes a vessel that is configured to contain a substrate. A compartment is provided as part of the system, the compartment being positioned to receive substrate from the vessel. The compartment is also positioned to return a portion of received substrate to the vessel during normal operation of the system.

The system also includes a filter that is positioned at least partially within the compartment and that is connected to withdraw permeate from substrate in the compartment during normal operation of the system. A source of cleaning solution is preferably connected to the compartment in order to introduce cleaning solution into the compartment and into contact with the filter during cleaning operation of the system.

In the system according to this invention, the compartment facilitates circulation of substrate during normal operation of the system. The compartment also makes it possible to substantially prevent the introduction of cleaning solution from the compartment into contact with substrate contained in the vessel during the cleaning operation of the system. Accordingly, the system of this invention is adapted for withdrawing permeate from a substrate through the filter and for cleaning the filter in situ in order to avoid the need for periodic removal of the filter.

A method is also provided for withdrawing permeate from a substrate through a filter and for cleaning the filter in situ. The method includes the steps of providing a compartment to at least partially surround the filter. During normal operation of the filter, substrate is introduced from a vessel into the compartment, permeate is withdrawn through the filter from substrate received in the compartment, and a portion of received substrate is returned from the compartment to the vessel. During cleaning operation of the filter, flow of substrate into the compartment is prevented, permeate is returned to the compartment, and a cleaning solution is preferably introduced into the compartment and into contact with the filter, all while maintaining the filter in situ.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. **1** is a block diagram of an embodiment of a system and method according to this invention.

FIG. **2** is a schematic diagram of another embodiment of a system and method according to this invention.

FIG. **3** is a schematic diagram of yet another embodiment of a system and method according to this invention.

DETAILED DESCRIPTION OF THE INVENTION

Features of this invention will now be described with reference to figures which illustrate selected embodiments of the invention. It will be appreciated that this invention is not limited to the embodiments selected for illustrated in the figures and that the scope of the invention is separately defined in the appended claims. It will also be appreciated that the figures are not drawn to any particular proportion or scale, and that the embodiments illustrated in the figures can be modified or varied without departing from the spirit or scope of this invention.

Features of this invention will now be described with reference to the block diagram provided in FIG. **1**. More specifically, FIG. **1** illustrates a system for withdrawing permeate from a substrate through a filter and for at least partially cleaning the filter in situ. The illustrated system **10** includes a vessel **12** that is configured to contain substrate. The composition of the substrate can vary while still achieving the benefits of this invention, but the substrate is most frequently a liquid or a slurry of liquid and solid particles.

System **10** also includes a compartment **14** connected to receive substrate from vessel **12**, wherein the flow of substrate from vessel **12** to compartment **14** is indicated at A in FIG. **1**. Compartment **14** is also connected to return a portion of received substrate to vessel **12** during normal operation of the system **10**. For example, the return flow of substrate from compartment **14** to vessel **12** is indicated at B in FIG. **1**. Compartment **14** also includes an opening for discharge from system **10**. More specifically, a discharge flow is indicated at C in FIG. **1**.

A filter **16** is positioned at least partially within compartment **14**. Filter **16** is connected to withdraw permeate from substrate in compartment **14** during normal operation of the system **10**. For example, permeate flow from filter **16** is indicated at Din FIG. **1**.

System **10** also includes a source **18** of cleaning solution. Source **18** is connected to introduce cleaning solution into compartment **14** and into contact with filter **16** during cleaning operation of system **10**. For example, the flow of cleaning solution from source **18** to compartment **14** is indicated at E in FIG. **1**.

The compartment **14** of system **10** facilitates the circulation of substrate through the system **10** during normal operation of the system. More specifically, substrate can be circulated by flow at A from vessel **12** to compartment **14** and by flow at B from compartment **14** to vessel **12**. Also, compartment **14** substantially prevents the introduction of cleaning solution, received from source **18**, from compartment **14** into contact with substrate contained in vessel **12**.

Still referring to FIG. **1**, an embodiment of a method according to this invention will now be described. System **10** illustrated in FIG. **1** is adapted for withdrawing permeate from a substrate through a filter and for at least partially cleaning the filter in situ. In use, compartment **14** is provided to at least partially surround filter **16**. During normal operation of system **10**, substrate is introduced from vessel **12** into compartment **14** in the form of flow at A. Permeate is withdrawn through filter **16** from substrate received in compartment **14** as indicated at D. Also during normal operation, a portion of received substrate is returned from compartment **14** to vessel **12** as indicated at B.

Cleaning operation of system **10** is illustrated in FIG. **1** by the use of dotted lines. More specifically, during cleaning

operation, flow of substrate into compartment **14** from vessel **12** as indicated at A is prevented. Permeate is returned to compartment **14** and/or through filter **16** as indicated at F. Cleaning solution is introduced at E from source **18** into compartment **14** and/or filter **16** and into contact with external surfaces of filter **16**. Cleaning solution may then be drained from compartment **14** as indicated at C, if necessary.

The method according to this invention is accomplished while maintaining the filter **16** in situ or in place with respect to compartment **14** and vessel **12**. In other words, filter **16** is maintained in place during the normal operation of system **10** and during cleaning operation of system **10**. Filter **16**, therefore, need not be removed from compartment **14** to accomplish a deep cleaning.

Referring now to FIG. **2**, a schematic diagram of another embodiment of a system and method according to this invention is provided. Like system **10**, system **100** is adapted for withdrawing permeate from substrate through a filter **116** and for at least partially cleaning the filter **116** in situ.

System **100** includes a vessel **112** configured to contain substrate. In this embodiment, substrate is introduced into vessel **12** via a feed H'. System **100** also includes a compartment **114** connected to receive substrate from vessel **112**. In this embodiment, substrate is delivered into compartment **114** from vessel **112** by means of a circulating pump **120** that urges substrate toward compartment **114** as indicated at A'. Compartment **114** is connected to return a portion of received substrate to vessel **112** during normal operation of the system.

In this embodiment, compartment **114** is positioned at least partially within vessel **112** and has an at least partially open top to permit the overflow of substrate from within compartment **114** into the interior of vessel **112** as indicated at B'. In order to prevent or reduce the tendency for concentration of biosolids in compartment **114**, a significant portion of substrate received in compartment **114** is intended to return as indicated at B' into vessel **112**. Preferably, the majority of substrate received in compartment **114** is returned to vessel **112**. Compartment **114** also includes an opening for discharge from system **100**. In this embodiment, a valve **126** is provided in order to control discharge flow as indicated at C'.

A filter **116** is positioned at least partially within, and preferably completely within, compartment **114**. Filter **116** is connected to withdraw permeate from substrate in compartment **114** during normal operation of the system. In this embodiment, a permeate pump **122** is connected to filter **116** in order to deliver permeate from filter **116** to a permeate tank **124** as indicated at D'.

A source **118** of cleaning solution is connected to introduce cleaning solution into compartment **114** and into contact with external surfaces of filter **116** during cleaning operation of system **100**. In this embodiment, source **118** delivers cleaning solution as indicated at E' so that it enters compartment **114** (and/or filter **116**) for contact with filter **116**.

Compartment **114** facilitates circulation of substrate through system **100** during normal operation of the system and substantially prevents the unintended introduction of cleaning solution from compartment **114** into contact with substrate contained within vessel **112** during cleaning operation.

Supplemental cleaning features can also be provided in system **100**. For example, permeate can be returned through filter **116** in order to provide periodic back pulsing of filter **116** in order to facilitate partial cleaning of filter **116**. Also, agitation air can be introduced proximal to filter **116** in order to cause the filter to vibrate and flex, although the source of such agitation air is not shown in FIG. **2**.

During use of system **100**, and during normal operation, substrate is introduced from vessel **112** into compartment **114** by means of circulating pump **120** as indicated at A'. Permeate is withdrawn through filter **116** from substrate received in compartment **114** and is delivered by means of permeate pump **122** to permeate tank **124** as indicated at D'. A portion of received substrate is returned from compartment **114** to vessel **112** as indicated at B'. The flow at B' is preferably greater than the flow at D'. Most preferably, the ratio of substrate flow at B' to the permeate flow at D' approaches or even exceeds 5:1.

Cleaning operation of system **100** is indicated by dotted lines. During cleaning operation of system **100**, flow of substrate into compartment **114** from vessel **112** is prevented (by deactivation of circulating pump **120**). Permeate is returned to compartment **114** and/or filter **116** from permeate tank **124** as indicated at F'. A cleaning solution is introduced from source **118** into compartment **114** (and/or filter **116**) and into contact with surfaces of filter **116** as indicated at E'. Cleaning solution is subsequently drained from compartment **114** by means of opening valve **126** to induce flow at C'. In order to assist in the cleaning operation, agitation air or other gas can be introduced adjacent to filter **116** as indicated at G'.

In the embodiment illustrated in FIG. **2**, a slurry of biosolids is circulated from a well-mixed reaction area within the vessel **112** through the compartment **114** at a rate equal to several times the permeate withdrawal rate. The excess biosolids slurry overflows the compartment **114**, thereby returning to the reaction area in the vessel **112**. The high rate preferred for overflow prevents undue concentration of biosolids in the compartment **114**. Fresh feed liquid is added to the reactor vessel **112** (at H') at a rate about equal to the rate at which it is being withdrawn as permeate.

When periodic chemical cleaning is required, the compartment **114** is isolated and drained of liquid or slurry (by means of valve **126**). The compartment **114** is then refilled with stored water previously processed through the membrane (from permeate tank **124**) along with the cleaning chemicals. As described, air or other agitation can be applied during the cleaning period as indicated at G'. Following the cleaning period, the cleaning solution can be drained from the compartment **114**, if necessary, and the compartment **114** can be refilled with biosolids liquid or slurry. The filter **116** can then be returned to normal operation.

This embodiment of the invention confers several significant benefits. Specifically, the filter does not have to be removed from the reactor vessel for cleaning. Accordingly, rigging equipment for filter removal is not required and the plumbing connections for the filter do not have to be disconnected/reconnected. System **100** also eliminates the need for an external cleaning tank in which to relocate the filter, thereby saving floor space and the associated plumbing. The time required for cleaning is accordingly reduced. Additionally, the introduction of the compartment, which at least partially surrounds the filter within the vessel, makes it unnecessary to discard or transfer a large volume of biosolids liquid or slurry or to provide a large volume of cleaning solution.

Referring now to FIG. **3**, a schematic diagram of yet another embodiment of a system and method according to this invention is illustrated. Like systems **10** and **100**, system **200** illustrated in FIG. **2** is adapted for withdrawing perme-

ate from substrate through a filter and for at least partially cleaning the filter in situ. Also, like system **100**, system **200** utilizes a vessel **212**, a compartment **214**, a filter in the form of a membrane cartridge **216**, a source of cleaning solution **218**, a circulating pump **220**, a permeate pump **222**, and a permeate tank **224**.

System **200** is provided with a feed source or substrate source **254**. Feed source **254** is connected to a line **256** through which feed or substrate is introduced into the interior of vessel **212**, as indicated in FIG. **3**. Substrate is delivered into compartment **214** from vessel **212** by means of a circulating pump **220**. More specifically, a line **202** extends from the wall of vessel **212** to circulating pump **220**, and lines **204** and **206** extend from circulating pump **220** to a location within compartment **214**, as will be described. A valve **208** is positioned along line **206** in order to control the flow of substrate between circulating pump **220** and compartment **214** through line **206**.

Positioned within compartment **214** is a diffuser pipe **210** having a series of openings to permit the flow of substrate from within diffuser pipe **210** into the interior of compartment **214**. Line **206** is connected to one end portion of diffuser pipe **210** in order to introduce substrate into the interior of diffuser pipe **210**. Diffuser pipe **210** is preferably a straight pipe that extends substantially horizontally with respect to the bottom surface of compartment **214**.

System **200** also includes means for delivering materials such as waste solids from compartment **214** or for draining compartment **214**. More specifically, a line **226** extends from an end of diffuser pipe **210** (an end opposite the end connected to line **206**) for the outflow from compartment **214** of waste solids as well as cleaning solutions, as will be described later. Line **226** is connected to a line **228** which, in turn, is connected to a waste solids receptacle **230**. A valve **232** positioned along line **228** controls the flow of materials from compartment **214** and diffuser pipe **210** through line **228** to the waste solids receptacle **230**.

A line **234** is also connected to line **226**, which is in turn connected to a compartment drain **236**. A valve **238** positioned along line **234** controls the flow of materials from compartment **214** and diffuser pipe **210** toward compartment drain **236** through line **234**.

Vessel **212** is considered to be a "well mixed tank" because it is provided with a mixing pump that brings about circulation of substrate in vessel **212**. The purpose is to keep biosolids suspended in the substrate during operation of system **200**. More specifically, a line **240** extends from the wall of vessel **212** to deliver substrate from vessel **212** to a mixing pump **242**. The mixing pump **242** urges substrate along a line **244** from line **240** so that it enters a mixer such as mixing eductor **248**. Mixing eductor **248** can be provided in the form of a module such as a pod with radially oriented nozzles such as the embodiment shown in FIG. **3**. Alternatively, mixing eductor **248** can be provided in the form of a pipe such as a straight pipe that extends at least partially across the diameter of vessel **212** with a series of outlet openings. The use of a straight pipe as opposed to a pod may be preferred for larger tanks that may have an extensive diameter. Other forms of an inlet such as eductor **248** are known in the art and can be substituted for the form illustrated in FIG. **3**. Also known in the art are other forms of mixing with or without the use of air or other gases.

System **200** also includes a source **250** of mix air or other gas that travels along line **252**, enters mixing eductor **248**, and is introduced into the interior of vessel **212**. The introduction of mix air into mixing eductor **248** for mixing with substrate creates agitation, which encourages the mixing of the substrate and the suspension of the biosolids within vessel **212**. The introduction of air, if air is used, also provides a source of oxygen to support the biological activity that occurs within vessel **212**.

System **200** also includes a membrane cartridge **216**, which extends at least partially, and preferably completely, within the interior of compartment **214**. The membrane cartridge of this embodiment is a submersible membrane filter having a series of hollow fiber membranes extending between manifolds. Permeate is extracted through membrane cartridge **216** by permeation through the walls of the hollow fiber membranes, transportation of the permeate through the membranes to the connected manifolds, and removal of permeate through a reduced-pressure piping system. More specifically, membrane cartridge **216** of system **200** has a bottom manifold **264**, a top manifold **266**, and a series of membranes such as hollow fiber membranes **268** extending substantially vertically between bottom manifold **264** and top manifold **266**. Fibers **268** provide a barrier through which permeate is drawn during normal operation of system **200** in order to extract permeate from the substrate.

As will be understood, when a vacuum is drawn at the interior of the fibers **268**, permeate is drawn through the walls of the hollow fibers **268** and into the interior of the hollow fibers **268** so that the permeate can be extracted via bottom and top manifolds **264** and **266** for extraction from the system **200**. In other words, by creating a pressure differential across the thickness of the walls of hollow fibers **268**, wherein the pressure on the outside of the fibers **268** is greater than the pressure within the interior of fibers **268**, permeate is caused to flow through pores in the walls of the hollow fibers **268** and into the interior thereof for extraction from system **200**. Biosolids such as bio-mass are blocked by the fibers **268** and remain in the compartment.

System **200** also includes a membrane air or other gas source **258** that introduces air or other gas into a line **260** so that it can be transported to an air manifold **262** that is positioned adjacent to or at least partially within membrane cartridge **216**. The air manifold **262** includes air outlets or nozzles (not shown) which permit the flow of air bubbles adjacent to the membranes **268** of membrane cartridge **216**. Such air bubbles can help to reduce the rate at which a film of bio-mass is formed on the outer surfaces of the membranes **268**. Air from membrane air source **258** also tends to encourage the mixing of substrate (and cleaning solution, as will be described) within compartment **214**.

Still referring to FIG. **3**, a pair of lines **270**, **272** extend upwardly from membrane cartridge **216**, wherein line **270** is connected to permit the flow of permeate upwardly from bottom manifold **264** and line **272** is connected to permit the flow of permeate upwardly from top manifold **266**. Lines **270** and **272** are connected to a line **274**, and a valve **276** is provided along line **274** in order to control the flow of permeate through line **274**.

Connected to line **274** is a line **278**, which is in turn connected to an air vent **280** for the ventilation of undissolved air from the permeate that may have been introduced into the permeate from an outside source such as membrane air source **258**. Also connected to line **274** is a line **282** that extends upwardly, and a permeate pump **222** is connected along line **282** in order to urge the flow of permeate through line **282**.

As an alternative to the use of permeate pump **222**, it has been discovered that gravity flow can be employed to

transfer permeate from membrane cartridge **216** to a permeate tank **224** (or directly to a discharge). More specifically, if the elevation of the membrane cartridge **216** is maintained above that of the permeate in permeate tank **224**, then permeate will flow from the membrane cartridge **216** to the permeate tank **224** by action of atmospheric pressure and a siphon effect. The configuration of compartment **214** and membrane cartridge **216** in the embodiment illustrated in FIG. **3** makes it possible, therefore, to eliminate permeate pump **222** and the energy required to run the pump if at least a portion of the permeate tank **224** is repositioned below the filter.

If "pulsed cleaning" is performed (as described later), it will be appreciated that a pump may be required to return permeate from permeate tank **224** to membrane cartridge **216** if the permeate tank **224** is positioned at an elevation below the filter. Such a pump would run less than permeate pump **222** because of the preferred intermittent nature of the "pulsed cleaning" operation (as described later) as compared to the substantially continuous running of permeate pump **222** during the cleaning operation.

Connected to line **282** is a line **284** on which a valve **286** is provided to control the flow through line **284**. Line **284** is, in turn, connected to a permeate tank **224**, which is adapted to contain and collect permeate extracted from the substrate in vessel **212**. Permeate within permeate tank **224** overflows into a baffle **225** from which the permeate or effluent is removed from the system **200** for use or for further processing.

Connected to the bottom portion of permeate tank **224** is a line **288** for delivering permeate toward line **282**. A valve **290** is provided along line **288** to control the flow of permeate from permeate tank **224** to line **282**. Also connected to line **282** is a line **292** for the flow of permeate from line **282** to lines **294** and **296**. Line **294** is provided with a valve **295** in order to control the flow of permeate therethrough, and line **296** is provided with a valve **297** to control the flow of permeate therethrough. Line **294** is connected between line **292** and line **274**. In contrast, line **296** is connected between line **292** and compartment **214** for the delivery of flow into compartment **214**.

The source of cleaning solution **218** is connected to line **292** by means of a line **298** that extends between source **218** and line **292** in order to permit the flow of cleaning solution from source **218** to the remainder of system **200**. Among other known cleaning solutions, chlorine solutions are preferred.

The preferred operation of system **200** will now be described with reference to FIG. **3**. Three general operations of the system will be described in terms of "normal operation" of system **200** during which permeate is extracted from the substrate, a "pulsed cleaning" of system **200** which preferably occurs periodically during normal operation, and a "deep cleaning" of system **200** which preferably occurs during an interruption of normal operation.

Generally, substrate is fed into vessel **212**, circulated to compartment **214**, and permeate is removed through membrane cartridge **216** during normal operation of system **200**. Permeate is delivered to permeate tank **224** or, alternatively, directly to discharge. At the same time, substrate is mixed in vessel **212** to maintain a well mixed tank.

During pulsed cleaning, which preferably occurs periodically during normal operation of system **200**, permeate is pulsed back into membrane cartridge **216** in a reverse flow direction (by pump or by atmospheric pressure) in order to help reduce the accumulation of biosolids or other organic or inorganic foulants on the surfaces of the fibers **268** of membrane cartridge **216**. Such a pulsed cleaning operation can be conducted at timed intervals for a short duration. For example, and for purposes of illustration only, pulsed cleaning can be conducted twice per hour, each time for a pre-determined duration of about one minute. Other intervals (more or less frequent) and durations (longer or shorter) are of course contemplated.

During deep cleaning, substrate contained in compartment **214** is drained and replaced with a cleaning solution for a predetermined period of time in order to bring about a chemical cleaning of the fibers **268** of membrane cartridge **216**. Thereafter, the cleaning solution can be drained and replaced with substrate from vessel **212**, if necessary, to return system **200** to normal operation.

Each of the normal, pulsed cleaning, and deep cleaning operations will be described hereafter in greater detail. With regard to normal operation, circulating pump **220** is actuated and valve **208** is opened so that substrate can be urged from vessel **212** into diffuser pipe **210** for introduction into the interior of compartment **214**. Valve **232** on line **228** can be periodically opened during normal operation in order to extract some of the biosolids that may otherwise accumulate within compartment **214**. During normal operation, valve **238** on line **234** is closed in order to prevent the flow of substrate from compartment **214** toward compartment drain **236**.

System **200** is designed so that the flow rate of substrate from vessel **212** into compartment **214** exceeds the flow rate of permeate from membrane cartridge **216** for removal from system **200**. Accordingly, the flow rate into compartment **214** of substrate through diffuser pipe **210** is greater than the flow rate of permeate out of compartment **214**. There will therefore be overflow of substrate over the upper edge or lip of compartment **214** into vessel **212**. For purposes of illustrating one preferred embodiment of this invention, if the flow rate of permeate from membrane cartridge **216** is "X", and if the flow rate of substrate from vessel **212** into compartment **214** is "6X", then the rate of overflow of substrate from compartment **214** back into vessel **212** will be approximately "5X" (6X−X=5X). It should be noted that some additional outflow of material will occur through valve **232** and into waste solids receptacle **230** during normal operation of the system. Accordingly, in the example provided, it would be expected that the overflow of substrate from compartment **214** back into vessel **212** would actually be slightly less than 5X.

It has been discovered that the overflow of excess substrate from the compartment back into vessel confers several benefits. Primarily, such overflow provides additional circulation to system **200**, thereby maintaining a more uniform suspension of bio-mass in the substrate. The overflow also helps to maintain the membrane cartridge within substrate in the compartment so that it remains completely submerged. The overflow also creates a flow pattern adjacent to the membrane cartridge so as to reduce the accumulation of bio-mass in the compartment and on the membrane surfaces.

Still during normal operation, permeate pump **222** is actuated and valves **276** and **286** are opened in order to draw permeate through the boundary provided by fibers **268**, into bottom and top manifolds **264** and **266**, through lines **270** and **272** to line **274**, through line **282** and line **284**, for delivery into the interior of permeate tank **224**. Alternatively, as described previously, permeate pump **222** need not be used if the elevations of the fibers **268** and permeate tank **224** are adjusted such that atmospheric pressure causes the permeate to flow from the fibers to the tank.

Permeate then exits permeate tank **224** through baffled area **225** for use or for further processing. Alternatively, permeate can be delivered directly to a discharge when the permeate tank is filled or it can completely bypass the permeate tank. During such normal operation, valves **290**, **295**, and **297** are closed (except as indicated below during pulsed cleaning) in order to prevent the return of permeate toward membrane cartridge **216**.

Also during normal operation of system **200**, feed of substrate is introduced from feed source **254**, through line **256**, and into vessel **212**. In order to maintain introduced substrate in a well mixed condition, mixing pump **242** is actuated, either periodically or continuously, in order to transfer substrate from vessel **212**, through lines **240** and **244**, and into mixing eductor **248**. At the same time, mix air is introduced from a source **250** of mix air or other gas, through line **252**, and into mixing eductor **248** to mix with the substrate. The nozzles on mixing eductor **248** deliver a mixture of substrate and mix air from mixing eductor **248** back into vessel **212** in order to maintain a well mixed tank.

Membrane air is also introduced during normal operation of system **200** from membrane air source **258**, through line **260**, through air manifold **262**, and into compartment **214** adjacent to the fibers **268** of membrane cartridge **216**. Membrane air thus introduced helps to provide agitation in the substrate adjacent to the fibers in order to reduce the tendency of bio-mass to settle in the form of a film on the surface of the fibers. Also during normal operation of system **200**, air is vented from line **274**, through line **278**, to air vent **280** in order to discharge undissolved air from the system.

As described above, pulsed cleaning is preferably conducted at predetermined intervals and for predetermined durations throughout the normal operation of system **200**. More specifically, at the designated intervals and for the designated duration, valves **290** and **295** are opened, and valves **276** and **286** are closed, so that permeate pump **222** (or atmospheric pressure as described above) can urge permeate from tank **224**; through lines **288**, **282**, **292**, **294**, **270**, and **272**; and into manifolds **264** and **266**, for flow into fibers **268**. This reverse flow causes permeate to flow in the opposite direction of normal operation through the fiber walls in such a manner as to reduce the build up of bio-mass on the outer walls of the fibers. As permeate is introduced along line **292**, it is preferably mixed with cleaning solution introduced from source **218** along line **298**.

The deep cleaning operation of system **200** will now be described, again with reference to the system **200** illustrated in FIG. **3**. Initially, to end normal operation of system **200**, the delivery of substrate from vessel **212** into compartment **214** is interrupted by deactivating circulating pump **220** and closing valve **208**. Substrate within compartment **214** is then drained by closing valve **232** and opening valve **238** so that the substrate in compartment **214** is drained along lines **226** and **234** into compartment drain **236**. This draining procedure is facilitated by the flow of substrate through the openings in diffuser pipe **210** for flow from the interior of diffuser pipe **210** into connected line **226**.

Valves **276**, **286**, and **295** are closed, and permeate pump **222** is actuated, in order to deliver permeate from permeate tank **224**; through lines **288**, **282**, **292**, and **296**; and into compartment **214**. Cleaning solution is simultaneously delivered from source **218** along line **298** to mix with the introduced permeate in line **292**. Compartment **214** is filled with a mixture of permeate and cleaning solution until it preferably reaches a height above the top manifold **266** of membrane cartridge **216** (so that the membrane cartridge will be fully submerged in the permeate/cleaning solution mixture) but below the upper lip of compartment **214** (so that the permeate/cleaning solution mixture will not flow over the edge of the compartment into the interior of vessel **212** for mixture with the substrate that is still within the interior of vessel **212**). Accordingly, the substrate within vessel **212** will not be contacted by a substantial amount of cleaning solution and, therefore, the cleaning solution will be prevented from attacking the bio-mass in the substrate, which could otherwise compromise the ability of the bio-mass to treat the substrate.

The membrane cartridge **216** is then "soaked" in the cleaning solution for a predetermined period of time in order to eliminate or reduce the amount of bio-mass that may have accumulated on the surfaces of the fibers **268** of the membrane cartridge **216**. Although various durations may be selected depending on the particular constituents of the substrate and biomass and other factors, the duration of the cleaning operation is preferably several hours and preferably as long as four hours or longer. Such "deep cleaning" may be advantageously performed once per month of normal operation or at more or less frequent intervals depending on the needs of the system and the rate at which a bio-film is generated on the fibers. In conjunction with the soaking of the membrane cartridge **216** in cleaning solution for the predetermined duration, membrane air can optionally be added from membrane air source **258** along line **260** and through air manifold **262** in order to provide additional agitation for the removal of bio-mass from the surface of the fibers **268**.

In addition to the addition of membrane air (or as an alternative to membrane air), the pulsed cleaning operation described previously can be performed during the cleaning operation in order to introduce permeate (with or without cleaning solution) into the interior of fibers **268** for reverse flow through membrane cartridge **216**. Such a combination of pulsed cleaning and deep cleaning can be advantageous to accelerate the elimination of bio-mass from the fiber surfaces.

After the selected duration of the cleaning cycle has elapsed, the valve **238** can be opened so that cleaning solution can be drained from compartment **214** through diffuser pipe **210**, lines **226** and **234**, for delivery to compartment drain **236**. After the cleaning solution has been drained from compartment **214**, the normal operation of system **200** (described above) can be restarted by once again introducing substrate from vessel **212** into compartment **214**.

Alternatively, if the cleaning solution is neutralized or consumed during the duration of the cleaning operation, then it is possible to proceed directly to normal operation of system **200** without draining compartment **214**. In other words, if the toxicity of the cleaning solution is degraded sufficiently during the cleaning operation so that it will not unduly inhibit the activity of the bio-mass, then substrate can simply be introduced into compartment **214** and into contact with the cleaning solution to bring about normal operation of system **200**. The spent cleaning solution is then diluted in the substrate for mixture in compartment **214** and vessel **212**. The ability to eliminate the draining step, whereby cleaning solution is drained from compartment **214** as described above, depends on the nature of the cleaning solution used, the volume of cleaning solution contained in compartment **214**, the constituents of the bio-mass, the duration of the cleaning operation, and other factors.

This invention has been described with reference to particular exemplary embodiments selected for illustration

in the drawings. It will be appreciated, however, that many variations and modifications of the embodiments selected for illustration can be made within the scope of the invention. The structure of the vessels and compartments illustrated schematically in FIGS. **2** and **3** can vary widely while maintaining the same function. The relative positioning of the compartment with respect to the vessel, whether the compartment is position wholly or partially within the vessel or outside the vessel, is not critical to the invention although the configurations depicted schematically in FIGS. **2** and **3** are preferred. The pipe and valve schemes diagramed in FIG. **3** can also be modified to be adapted to a particular use or a particular system. The type of filter used to withdraw permeate can vary even though preferred embodiments of the invention have been described with reference to submersible filters such as hollow fiber membranes.

Additional modifications and variations can be made without departing from the spirit or scope of the invention. The invention is defined separately in the appended claims.

What is claimed:

1. A system for withdrawing permeate from a substrate through a filter during operation of the system and for at least partially cleaning the filter in situ during cleaning of the system, said system comprising:

a vessel configured to contain substrate;

a compartment configured to receive substrate from said vessel and to return a portion of received substrate to said vessel during operation of the system;

a filter positioned at least partially within said compartment and configured to separate permeate from substrate in said compartment during operation of the system;

said system being configured for facilitating circulation of substrate during operation of the system and for containing cleaning solution in said compartment and substantially preventing cleaning solution from contacting substrate in said vessel during cleaning of the system.

2. The system recited in claim **1**, further comprising a source of cleaning solution configured to introduce cleaning solution into said compartment and into contact with said filter during cleaning of the system.

3. The system recited in claim **1**, said compartment having an opening for discharge of cleaning solution or substrate from the system.

4. The system recited in claim **1**, further comprising a diffuser positioned within said compartment for receiving substrate delivered from said vessel and for introducing received substrate into said compartment.

5. The system recited in claim **1**, said filter comprising a membrane through which permeate is separated during operation of the system.

6. The system recited in claim **5**, said membrane comprising a plurality of hollow fibers.

7. The system recited in claim **1**, said compartment defining an opening through which received substrate returns to said vessel.

8. The system recited in claim **7**, said opening being positioned at a top portion of said compartment.

9. The system recited in claim **1**, said filter being configured to be submerged in substrate during operation.

10. The system recited in claim **9**, said filter being positioned completely within an interior of said compartment.

11. The system recited in claim **1**, further comprising a tank connected to receive permeate separated by said filter.

12. The system recited in claim **1**, further comprising a permeate discharge positioned at an elevation below said filter such that atmospheric pressure causes permeate to flow from said filter toward said permeate discharge.

13. A system for withdrawing permeate from a substrate through a filter during operation of the system and for at least partially cleaning the filter in situ during cleaning of the system, said system comprising:

a vessel configured to contain substrate;

a compartment configured to receive substrate from said vessel and to return a portion of received substrate to said vessel during operation of the system;

a filter positioned at least partially within said compartment and configured to separate permeate from substrate in said compartment during operation of the system;

said system being configured for facilitating circulation of substrate during operation of the system and for containing cleaning solution in said compartment and substantially preventing cleaning solution from contacting substrate in said vessel during cleaning of the system, said compartment being positioned at least partially within said vessel.

14. A method for withdrawing permeate from a substrate through a filter during operation and for at least partially cleaning the filter in situ during cleaning, said method comprising the steps of:

(a) providing a compartment at least partially surrounding the filter;

(b) during operation,

(i) introducing substrate from a vessel into the compartment,

(ii) returning a portion of received substrate from the compartment to the vessel, and

(iii) withdrawing, through the filter, permeate from substrate received in the compartment; and

(c) during cleaning,

(i) preventing flow of substrate into the compartment from the vessel,

(ii) introducing a cleaner into the compartment or filter, and

(iii) at least partially submerging the filter to at least partially clean the filter, all while maintaining the filter in situ.

15. The method recited in claim **14**, wherein the cleaner introducing step comprises introducing permeate, a chemical solution, or a combination of permeate and a chemical solution.

16. The method recited in claim **14**, wherein the providing step includes positioning the filter completely within the interior of the compartment.

17. The method recited in claim **14**, said returning step including returning a majority of received substrate from the compartment to the vessel.

18. The method recited in claim **14**, further comprising the step, during operation, of maintaining the ratio of returned substrate to permeate at about 5:1.

19. The method recited in claim **14**, further comprising the step of mixing substrate in the vessel.

20. The method recited in claim **14**, further comprising the step, during cleaning, of draining cleaner from the compartment.

21. The method recited in claim **14**, wherein operation and cleaning are alternated periodically.

22. The method recited in claim **14**, said returning step causing circulation of received substrate adjacent to the filter to reduce the formation of a film on the filter.

23. The method recited in claim **14**, further comprising the step, during cleaning, of returning to the filter a portion of permeate for reverse flow through the filter.

24. The method recited in claim **23**, said step of returning permeate to the filter being performed periodically.

25. The method recited in claim **14**, wherein the cleaner introducing step comprises introducing cleaner into the compartment through the filter.

26. A method for withdrawing permeate from a substrate through a filter during operation and for at least partially cleaning the filter in situ during cleaning, said method comprising the steps of:

a) providing a compartment at least partially surrounding the filter and positioning the compartment at least partially within the vessel;

(b) during operation,

(i) introducing substrate from a vessel into the compartment,

(ii) returning a portion of received substrate from the compartment to the vessel, and

(iii) withdrawing, through the filter, permeate from substrate received in the compartment; and

(c) during cleaning,

(i) preventing flow of substrate into the compartment from the vessel,

(ii) introducing a cleaner into the compartment or filter, and

(iv) at least partially submerging the filter to at least partially clean the filter, all while maintaining the filter in situ.

* * * * *

RELATED PROCEEDINGS APPENDIX

1. Appeal 2006-2492, decided on February 16, 2007, in the present Application Serial No. 09/916,247.

2. Appeal 2007-0362, decided on March 23, 2007, in Application Serial No. 09/425,234.

3. Appeal 2006-2898, decided on February 28, 2007, in Application Serial No. 10/461,687.

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS
AND INTERFERENCES

Ex parte PIERRE COTE, HAMID RABIE, NICHOLAS ADAMS, HIDAYAT HUSAIN, HENRY BEHMANN, STEVEN PEDERSEN, and JASON CADERA

Appeal 2006-2492
Application 09/916,247
Technology Center 1700

Decided: February 16, 2007

Before BRADLEY R. GARRIS, CHUNG K. PAK, and JEFFREY T. SMITH, *Administrative Patent Judges*.

SMITH, *Administrative Patent Judge*.

DECISION ON APPEAL

This appeal involves claims 26-36, the only claims pending in this application. We have jurisdiction under 35 U.S.C. § 134.

BRIEF STATEMENT OF THE INVENTION

Appellants' invention is directed to a process for filtering water containing solids with the membrane in a tank. The claimed process includes the backwashing with a chemical cleaner one or more suction driven ultrafiltration or microfiltration membranes normally immersed in water containing solids that become dirty or fouled during normal operation. Representative independent claim 26, as presented in the Brief, appears below:

> 26. A process for filtering water containing solids with membranes in a tank comprising the steps of:
>
> a) filling the tank with a feed water to be filtered to immerse the membranes;
>
> b) creating a transmembrane pressure between a permeate side and a retentate side of the membranes, the retentate side of the membranes being in contact with the water in the tank at ambient pressure, the permeate side being subject to a negative pressure relative to the pressure of the water in the tank fluidly connected to a filtered permeate outlet, to generate a filtered permeate at the permeate outlet and a retentate in the tank;
>
> c) aerating the membranes to dislodge solids from the membranes;
>
> d) backwashing the membranes; and,
>
> e) draining the tank of the retentate;
>
> wherein
>
> i) the steps above are performed in repeated cycles; and,

ii) the steps of backwashing the membranes and draining the tank in a cycle may be performed either before the other or partially or substantially simultaneously; and,

f) wetting the membranes at least once per week with a cleaning chemical having a selected concentration for a selected duration after performing step (b) in a first cycle and after or while performing step (e) in the first cycle, without returning to step (b) in the first cycle and before starting a subsequent cycle.

The Examiner relies on the following references in rejecting the appealed subject matter:

Smith	US 5,403,479	Apr. 4, 1995
Del Vecchio	US 6,331,251 B1	Dec. 18, 2001

U.S. Application 11/106,681 for double patenting.

The Examiner entered the following final rejections:

I. Claims 26-36 are rejected under 35 U.S.C. § 102(b) as anticipated by Smith.

II. Claims 26-28, 31, and 33-36 are rejected under 35 U.S.C. § 102 (e) as anticipated by Del Vecchio.

III. Claims 29, 30, and 32 are rejected under 35 U.S.C. § 103 as obvious over the combined teachings of Del Vecchio and Smith.

IV. Claims 26-29, 31, and 33 are provisionally rejected for a statutory double patenting over the copending claims 1-6 of application 11/106,681.

V. Claims 26-36 are provisionally rejected for obviousness-type double patenting over the copending claims 7-29 of application number 11/106,681.

I. Claims 26-36 are rejected under 35 U.S.C. § 102 (b) as anticipated by Smith. [1]

ISSUE

The Examiner contends that Smith figure 4 describes a process of filtering water containing solids that includes backwashing the membrane at least once a week with a cleaning fluid of selected concentration (Answer 5). The Examiner recognizes that Smith does not describe the backwashing of the filter system in the discussion of figure 4. However, the Examiner contends that Smith provides a discussion in the "background of the invention" portion of the specification that teaches draining the tank is not necessary during the cleaning process (Answer 5).

The issue before us is whether the Examiner has properly determined that the Smith reference teaches or describes the claimed subject matter under 35 U.S.C. §102(b). Specifically, the issue is whether the Examiner has properly determined that Smith describes a specific disclosure that includes the backwashing of the membranes, draining the tank of retentate, and wetting the membranes at least once per week with a cleaning chemical? We answer this question in the negative.

PRINCIPLES OF LAW

The Examiner bears the initial burden of establishing a prima facie case of anticipation *In re King*, 801 F.2d 1324, 1326-27, 231 USPQ 136, 138 (Fed. Cir. 1986). Anticipation under 35 U.S.C. § 102 requires that "each and every element as set forth in the claim is found, either expressly or inherently described, in a single prior art reference." *In re Robertson* 169 F.3d 743, 745, 49 USPQ2d 1949, 1950 (Fed. Cir. 1999).

[1] We will limit our discussion to claim 26, the only independent claim presented in the rejection.

Inherency may not be established by probabilities or possibilities, i.e., the mere fact that a certain thing may result from a given set of circumstances is not sufficient. *In re Oelrich*, 666 F.2d 578, 581, 212 USPQ 323, 326 (CCPA 1981).

ANALYSIS

The Examiner has specifically identified Figure 4 as describing the periodic cleaning of the filter system described by Smith. The Smith reference description of Figure 4 appearing in column 18 does not include a description of draining the tank during the cleaning process. The Examiner direct us to column 10 and for describing an alternative embodiment that indicates draining the tank is not necessary during the cleaning process (Answer 5). The Examiner subsequently states that "a reference may be relied upon for all that it would have reasonably *suggested* to one having ordinary skill in the art, including nonpreferred embodiments" (Answer 5) (emphasis added). The Examiner has not carried the burden of making out a prima facie case of anticipation in the first instance by pointing out where each and every element of the claimed invention, arranged as required by the claim, is described identically in the reference, either expressly or under the principles of inherency. It appears the Examiner is relying on suggestions of the reference to assert the requirements of the present invention are inherently possessed by the Smith reference. However, inherency cannot be based upon probabilities or possibilities. Suggestions and inferences which could have been derived from a reference are not proper basis for formulating an anticipation rejection under § 102. The stated rejection is reversed.

II. Claims 26-28, 31, and 33-36 are rejected under 35 U.S.C. § 102 (e) as anticipated by Del Vecchio.[2]

ISSUE

The Examiner contends that Del Vecchio describes a process of filtering water containing solids that includes backwashing the membrane at least once a week with a cleaning fluid (Answer 8). The Examiner contends that Del Vecchio describes the frequency of cleaning cycles of at least one week in column 12 of the reference. Specifically, the reference states "such 'deep cleaning' may be advantageously performed once per month of normal operation or at more or less frequent intervals depending on the needs of the system and the rate at which a bio-film is generated on the fibers." (Col. 12, ll. 20-24).

The issue before us is whether the Examiner has properly determined that the Del Vecchio reference teaches or describes the claimed subject matter under 35 U.S.C. § 102(b). Specifically, the issue is whether the Examiner has properly determined that Del Vecchio describes the cleaning frequency of at least one week as specified in claim 26? We answer this question in the negative.

PRINCIPLES OF LAW

The Examiner bears the initial burden of establishing a prima facie case of anticipation *In re King*, 801 F.2d 1324, 1326-27, 231 USPQ 136, 138 (Fed. Cir. 1986). Anticipation under 35 U.S.C. § 102 requires that "each and every element as set forth in the claim is found, either expressly or inherently described, in a single prior art reference." *Robertson* 169 F.3d at 745, 49 USPQ2d at 1950.

[2] We will limit our discussion to claim 26, the only independent claim presented in the rejection.

Inherency may not be established by probabilities or possibilities, i.e., the mere fact that a certain thing may result from a given set of circumstances is not sufficient. *Oelrich*, 666 F.2d at 581, 212 USPQ at 326.

ANALYSIS

The Examiner has specifically identified column 12 as describing the periodic cleaning of the filter system. The relevant portion of the Del Vecchio reference identified by the Examiner does not specifically describe the frequency of at least once a week as specified by the claimed invention. The description of Del Vecchio, that the cleaning operation can occur more or less frequently does not indicate that the claimed cleaning interval is expressly or inherently described in the disclosure of the reference. At best, the description of Del Vecchio relied upon by the Examiner would suggest to a person of ordinary skill in the art that the cleaning interval can be varied. Inferences and suggestions derived from a reference is not an indication that the claimed property or condition is expressly or inherently possessed by the reference. The stated rejection is reversed.

III. Claims 29, 30, and 32 are rejected under 35 U.S.C. § 103 as obvious over the combined teachings of Del Vecchio and Smith.

The rejected claims depend upon claim 26, which has been discussed above in the § 102 rejections. The Examiner has not provided an obviousness analysis of claim 26. The Examiner also has not provided an analysis of claim 26 in the discussion of this rejection. The Examiner bears the initial burden of establishing a prima facie case of obviousness. *In re Kumar*, 418 F.3d 1361, 1366, 76 USPQ2d 1048, 1051 (Fed. Cir. 2005). To support a rejection on obviousness grounds, the Examiner must provide a detailed analysis of the prior art and reasons why one of ordinary skill in the art would have possessed the knowledge and motivation to make the claimed

invention. *See In re Kahn*, 441 F.3d 977, 987-88, 78 USPQ2d 1329, 1336 (Fed. Cir. 2006). Since the Examiner has failed to provide a proper analysis of independent claim 26 under § 103 we *vacate* the Examiner's rejection of claims 29, 30, and 32 and return the application to the Examiner to provide a proper analysis of the rejected claims over the Smith and/or Del Vecchio references. The Examiner should also consider whether Smith and/or Del Vecchio affect the patentability of the other claims on appeal under § 103.

Double Patenting Rejections

IV. Claims 26-29, 31, and 33 are provisionally rejected for a statutory double patenting over the copending claims 1-6 of application 11/106,681.

V. Claims 26-36 are provisionally rejected for obviousness-type double patenting over the copending claims 7-29 of application number 11/106,681.

Appellants do not dispute that the appealed claims are patentably indistinct from the claims of the copending application 11/106,681. Rather, Appellants contend that the double patenting rejections are provisional and should be withdrawn in the present application and converted into the non-provisional rejections in the 11/106,681 application (Br. 4). Appellants citation to *the Manual of Patenting Examining Procedure* (*MPEP*) § 804, part IB, does not provide a basis for withdrawing the rejections in the present application, because these are not the sole rejections remaining in the present case. Appellants have not substantively challenged the merits of the stated rejections. We therefore uphold with the Examiner's rejections.

ORDER

The rejection of claims 26-36 under 35 U.S.C. §102(b) as anticipated by Smith is reversed.

The rejection of claims 26-28, 31, and 33-36 under 35 U.S.C. §102(e) as anticipated by Del Vecchio is reversed.

The rejection of claims 29, 30, and 32 are rejected under 35 U.S.C. §103 as obvious over the combined teachings of Del Vecchio and Smith is vacated and returned to the Examiner for proper analysis of the independent claim and other dependent claims under § 103.

The provisional rejection of claims 26-29, 31, and 33 for a statutory double patenting over the copending claims 1-6 of application 11/106,681, is affirmed.

The provisional rejection of claims 26-36 for obviousness-type double patenting over the copending claims 7-29 of application number 11/106,681, is affirmed.

We remand the application to the Examiner for proper determination of whether the subject matter of claims 26-36 is obvious within the meaning of 35 U.S.C. § 103(a) over the Smith and/or Del Vecchio references individually or combined.

In addition to affirming the Examiner's rejection of one or more claims, this decision contains a remand. 37 C.F.R. § 41.50(e) (2004) provides that

> [w]henever a decision of the Board includes a remand, that decision shall not be considered final for judicial review. When appropriate, upon conclusion of proceedings on remand before the examiner, the Board may enter an order otherwise making its decision final for judicial review.

Regarding any affirmed rejection, 37 C.F.R. § 41.52(a)(1) provides "Appellant may file a single request for rehearing within two months of the date of the original decision of the Board."

The effective date of the affirmance is deferred until conclusion of the proceedings before the Examiner unless, as a mere incident to the limited proceedings, the affirmed rejection is overcome. If the proceedings before the Examiner do not result in allowance of the application, abandonment or a second appeal, this case should be returned to the Board of Patent Appeals and Interferences for final action on the affirmed rejections, including any timely request for rehearing thereof.

AFFIRMED and REMANDED

hh

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The opinion in support of the decision being entered today was *not* written for publication and is *not* binding precedent of the Board.

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES

Ex parte HAMID RABIE, HIDAYAT, HUSAIN and HENRY BEHMANN

Appeal 2007-0362
Application 09/425,234
Technology Center 1700

Decided: March 23, 2007

Before BRADLEY R. GARRIS, CHUNG K. PAK, and JEFFREY T. SMITH, *Administrative Patent Judges*.

SMITH, *Administrative Patent Judge*.

DECISION ON APPEAL

This appeal involves claims 5-17, the only claims pending in this application. We have jurisdiction under 35 U.S.C. § 134.

BRIEF STATEMENT OF THE INVENTION

Appellants' invention is directed to a method of chemical cleaning one or more ultrafiltration or microfiltration membranes, normally immersed in water containing solids, that become dirty or fouled during normal operation. Representative independent claim 5, as presented in the Brief, appears below:

> 5. A method of cleaning one or more membranes normally immersed in water containing solids in a tank, the one or more membranes arranged into one or more modules such that permeate sides of the one or more membranes enclose a space in communication with one or more headers of the one or more modules, and used to produce a filtered permeate comprising:
>
> performing cleaning events having the steps of:
>
> (a) stopping permeation;
>
> (b) after step (a), and before resuming permeation, flowing a chemical cleaner to the one or more headers in a series of pulses, wherein the pulses are separated from each other by waiting periods in which the flow of chemical cleaner is stopped;
>
> (c) after step (b), resuming permeation;
> wherein
>
> (d) the membranes remain immersed in the water containing solids while the chemical cleaner flows to the one or more headers;
> (e) the outside of the membranes is in fluid communication with the water containing solids; and
>
> (f) during step (b), all chemical cleaner reaching the one or more headers remains in the enclosed space of the one or more modules or flows through the walls of the membranes in a direction opposite to the direction in which permeate normally passes through the walls of the membranes.

The Examiner relies on the following reference in rejecting the appealed subject matter:

Smith	US 5,403,479	Apr. 4, 1995

The Examiner entered the following final rejections:

I. Claims 6-10 are rejected under 35 U.S.C. § 112, second paragraph, as indefinite for failing to particularly point out and distinctly claim the subject matter which is regarded as the invention.

II. Claims 5-10 and 13-17 are rejected under 35 U.S.C. § 102 (b) as anticipated by Smith.

III. Claims 11 and 12 are rejected under 35 U.S.C. § 103(a) as unpatentable over Smith.

IV. Claims 5-17 are rejected for obviousness type double patenting over the copending claims 1-23 of application 11/106,681.

DISCUSSION

I. Claims 6-10 are rejected under 35 U.S.C. § 112, second paragraph, as indefinite for failing to particular point out and distinctly claim the subject matter which is regarded as the invention.

The Examiner contends that the limitation "more intensive first cleanings" indefinite since it is not defined in the present specification (Answer 4). Appellants contend the phrase "more intensive first cleanings" was introduced by amendment on January 14, 2004. Appellants assert that since the introduction of this claim language, there have been several office actions and the Examiner understood the claim well enough to examine it (Br. 6).

"The legal standard for definiteness [under the second paragraph of 35 U.S.C. § 112] is whether a claim reasonably apprises those of skill in the art of its scope." *In re Warmerdam*, 33 F.3d 1354, 1361, 31 USPQ2d 1754, 1759 (Fed. Cir. 1994). The inquiry is to determine whether the claim sets out and circumscribes a particular area with a reasonable degree of precision and particularity. The definiteness of the language employed in a claim must be analyzed not in a vacuum, but in light of the teachings of the particular application. *In re Moore*, 439 F.2d 1232, 1235, 169 USPQ 236, 238 (CCPA 1971). This is especially true in a situation involving a relative claim expression since the specification must provide some standard for defining or measuring its meaning. *Seattle Box Co. v. Industrial Crating & Packing, Inc.*, 731 F.2d 818, 826, 221 USPQ 568, 574 (Fed.Cir. 1984).

After consideration of the present record, we determine that a person of ordinary skill in the art would not have recognized the scope of the disputed relative claim language. Appellants have not directed us to specific portions of the Specification, from which a clear meaning of the phrase could be gleaned. That is, we have not been directed to portions of the Specification that provide guidance to determine the meaning of the claimed "more intensive" cleaning. As such, the Examiner's rejection on this basis is affirmed.

II. Claims 5-10 and 13-17 are rejected under 35 U.S.C. § 102 (b) as anticipated by Smith.

The Examiner contends that Smith describes a method for cleaning one or more membranes normally immersed in water containing solids. The Examiner contends the method comprises stopping the flow of the cleaning chemicals by pulsing wherein a low pressure above atmospheric, but no

more than the bubble point of the membrane is cyclically used to clean the headers/lumen (Answer 5 and 10). Appellants contend that the range of pressures described in the Smith reference provides the minimum and maximum range for the low pressure part of the cycle. Specifically Appellants contend that the reference describes decreasing the pulse rate, but does not imply stopping the flow between pulses (Br. 7).

The Examiner contends that Smith, (col. 11 ll. 29-47; and col. 17 ll. 50-56), describes the non-recirculating flow of chemical cleaner during the pulse cleaning cycles. On the other hand, the Appellants contend that Smith does not disclose dead-ending the cleaning chemical (Br. 7-8).

The issue before us is whether the Examiner has properly determined that the Smith reference teaches or describes the claimed subject matter under 35 U.S.C. § 102(b). Specifically, the first issue is whether the Smith reference describes flowing a chemical cleaner to one or more headers in a series of pulses, wherein the pulses are separated from each other by waiting periods in which the flow of the chemical cleaner is stopped. The second issue before us is whether the Smith reference describes the non-recirculating (dead end) flow of chemical cleaner through the walls of the membrane separated by pulses.

Smith describes a method of cleaning one or more membranes normally immersed in water containing solids that comprises introducing cleaning fluid into the permeate and recycling in through the lumens at a low pressure from about atmospheric, but below the bubble point of the fiber. Specifically Smith states:

> Highly effective cleaning of a module containing an UF or a MF membrane having a fouled surface is obtained during an unexpectedly short period, without draining feed (substrate) from

> the module, by introducing a chosen cleaning fluid into the permeate and recycling it through the lumens at low pressure in the range from about atmospheric but no more than the bubble-point of the fiber. The method comprises maintaining a selected low pressure no more than the bubble-point either continuously, or cyclically applied, over a short period of time, preferably less than 1 hr, sufficient to diffuse enough cleaning fluid through pores in the membrane into the dirty water, substantially to re-establish the initial stable flux. The low pressure may be substantially constant, or it may be deliberately varied within a period of less than 5 sec, preferably less than 1 sec. When pulsed to achieve pulsed diffusion, the pressure exerted by the cleaning fluid may vary from a minimum of about 100 kPa (1 bar, at least 0.1 psig, preferably 0.5 psig) for a "loose" MF (5μm) to a maximum of 100 psig for a "tight" UF (50Å), within less than 1 sec, which pulsing affords diffusion-controlled permeation. [Col. 11, ll. 21-43.]

Smith also discloses (col. 16, l. 68 to col. 17, l. 2) that:

> Since there is very little hydraulic pressure, typically less than 1.33 bar (5 psig) exerted by the cleaning fluid in the pores of the membrane while the fluid is recirculating through the membrane, and insufficient pressure to cause hydraulic flow of solution through the pores eve if pulsed, the flux obtained with the solution, is essentially diffusion-controlled and foulant lodged in the pores cannot be dislodged by hydraulic pressure. Instead, foulants are dissolved by chemical action. The main purpose of pulsing is to avoid, to the extend possible, diffusion flow through pores....

We agree with Examiner that the above passages in the Smith reference describe the pulse cycling of the cleaning fluid. The Examiner also correctly asserts that all of the pressures taught by Smith are below the bubble point and the peak pressures of the pulses are the bubble point. Appellants also acknowledge that all the pressures of the Smith reference are below the bubble point of the membranes (Reply Br. 2-3). It follows that

substantial evidence supports the Examiner' finding that there would have been no flow through the membrane except at the peak pressures, i.e., the pulsing of the cleaning fluid.

Appellants argue that the bubble point pressure only refers to the flow of gas as a bubble breaking through a pore and that gas can flow by diffusion through a pore at less than the bubble point pressure (Reply Br. 3). Appellants' arguments are not persuasive. They are not supported by any objective evidence. *In re De Blauwe*, 736 F.2d 699, 705,222 USPQ 191, 196 (Fed. Cir. 1984).

Regarding the second issue before us, the Examiner cites Smith (col. 11 ll. 29-47; and col. 17 ll. 50-56) for describing the non-recirculating flow of chemical cleaner during the pulse cleaning cycles (Answer 1). Smith, for example, discloses (col. 17, ll. 50-56) that:

> Check valve 23 is left openwhen cleaning solution is either circulated with pump 24 or pulsed when a pulse pump is substituted for pump 24. In those instances where it is desired to "dead end" the biocidal solution under only enough pressure to permit its diffusion-controlled flow out of the fibers, both the check valves 26 and 28 are closed.

Appellants' response appearing on page 3 of the Reply Brief does not explain why the cited portions of the Smith reference does not describe dead end flow as asserted by the Examiner. After review of the cited portions of the Smith reference, we agree with the Examiner's position.

We procedurally reverse the rejection of claims 6 to 10 over the Smith reference. Claims 6 to 10, have been rejected under 35 U.S.C. § 102(b) as unpatentable over Smith. We have carefully considered the subject matter defined by these claims, however, for reasons stated supra in our discussion

of the rejection under the second paragraph of 35 U.S.C. § 112, no reasonably definite meaning can be ascribed to certain language appearing in the claims. As the court in *In re Wilson*, 424 F.2d 1382, 1385, 165 USPQ 494, 496 (CCPA 1970) stated:

> [a]ll words in a claim must be considered in judging the patentability of that claim against the prior art. If no reasonably definite meaning can be ascribed to certain terms in the claim, the subject matter does not become obvious-the claim becomes indefinite.

In comparing the claimed subject matter with the applied prior art, it is apparent to us that considerable speculations and assumptions are necessary in order to determine what in fact is being claimed. Since a rejection based on prior art cannot be based on speculations and assumptions, see *In re Steele*, 305 F.2d 859, 862, 134 USPQ 292, 295 (CCPA 1962), we are constrained to reverse, pro forma, the Examiner's prior art rejections of claims 6 to 10. We hasten to add that this is a procedural reversal rather than one based upon the merits of the prior art rejections, as noted above.

III. Claims 11 and 12 are rejected under 35 U.S.C. § 103(a) as unpatentable over Smith.

Claims 11 and 12 define waiting periods between the claimed cycles of pulses. Appellants have not disputed the Examiner's finding that such periods are no more than result effective variables. Compare Answer 9 with Br. 10-11. Nor have Appellants proffered any evidence of unexpected results for the claimed subject matter . Thus, we agree with the Examiner's determination that the subject matter of claims 11 and 12 would have been obvious. .

IV. Claims 5-17 are rejected for obviousness-type double patenting over the copending claims 1-23 of application 11/106,681.

Appellants do not dispute that the appealed claims are patentably indistinct from the claims of the copending application 11/106,681. Rather, Appellants contend that the double patenting rejections are provisional and should be withdrawn in the present application and converted into the non-provisional rejections in the 11/106,681 application (Br. 5). Appellants citation to *the Manual of Patenting Examining Procedure* (*MPEP*) § 804, part IB, does not provide a basis for withdrawing the rejections in the present application, because these are not the sole rejections remaining in the present case. Appellants have not substantively challenged the merits of the stated rejections. We therefore uphold with the Examiner's rejections.

CONCLUSION OF LAW

The Examiner did not err in rejecting claims 6-10 under 35 U.S.C. § 112, second paragraph.

The Examiner did not err in rejecting claims 5 and 13-17 under 35 U.S.C. § 102(b) as anticipated by Smith.

The Examiner did not err in rejecting claims 11-12 under 35 U.S.C. § 103 as obvious over Smith.

The Examiner did not err in provisionally rejecting claims 5-17 for obviousness type double patenting over the copending claims 1-23 of application 11/106,681.

The Examiner erred in rejecting claims 6-10 under 35 U.S.C. § 102(b) as anticipated by Smith.

ORDER

The rejection of claims 6-10 under 35 U.S.C. § 112, second paragraph, is affirmed.

The rejection of claims 5 and 13-17 under 35 U.S.C. § 102(b) as anticipated by Smith is affirmed.

The rejection of claims 11-12 under 35 U.S.C. § 103(a) as obvious over Smith is affirmed.

The provisional rejection of claims 5-17 for obviousness type double patenting over the copending claims 1-23 of application 11/106,681, is affirmed.

The rejection of claims 6-10 under 35 U.S.C. § 102(b) as anticipated by Smith is reversed.

No time period for taking any subsequent action in connection with this appeal maybe extended under 37 C.F.R. § 1.136(a)(1)(iv) (2007).

AFFIRMED

tf/hh

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UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS
AND INTERFERENCES

Ex parte HAMID R. RABIE, HIDAYAT HUSAIN,
and HENRY BEHMANN

Appeal 2006-2898
Application 10/461,687
Technology Center 1700

Decided: February 28, 2007

Before BRADLEY R. GARRIS, CHUNG K. PAK, and
JEFFREY T. SMITH, *Administrative Patent Judges*.

SMITH, *Administrative Patent Judge*.

DECISION ON APPEAL

This appeal involves claims 1, 3, 12-16, 35, 36, and 41-43, the only claims pending in this application. We have jurisdiction under 35 U.S.C. § 134.

We REVERSE.

BRIEF STATEMENT OF THE INVENTION

Appellants' invention is directed to a method of chemical cleaning one or more ultrafiltration or microfiltration membranes normally immersed in water containing solids that become dirty or fouled during normal operation. Representative independent claims 1 and 35, as presented in the Brief, appear below:

1. A method for cleaning one or more membranes normally immersed in water containing solids held at ambient pressure in a tank and used to produce a permeate on the insides of the membranes, comprising the steps of:

(a) cleaning the membranes to increase the permeability of the membranes,

(b) after step (a), performing one or more cleaning events per week over a period of at least 15 days, each cleaning event further comprising the steps of flowing a chemical cleaner through the membranes to provide a chemical cleaner in or adjacent the membranes for a period of time; and,

(c) choosing the concentration of the chemical cleaner and the period of time such that the cleaning events reduce the rate of a decline in permeability of the membranes over the period of at least 15 days;

wherein steps (a) and (b) are preformed in repeated cycles.

35. A method for cleaning one or more hollow fiber membranes normally immersed in water containing solids held at ambient pressure in a tank and used to produce a permeate on the insides of the membranes, comprising the steps of:

(a) performing recovery cleanings to increase the permeability of the membranes from time to time; and

(b) between recovery cleanings, performing one or more cleaning events per week, each cleaning event comprising the steps of flowing a chemical cleaner through the membranes to provide a chemical cleaner in or adjacent the membranes for a period of time.

The Examiner relies on the following reference in rejecting the appealed subject matter:

Smith	US 5,403,479	Apr. 4, 1995

The Examiner rejected claims 1, 3, 35, and 43 under 35 U.S.C. § 102(b) as anticipated by Smith.

The Examiner rejected claims 12-16, 36, 41, and 42 under 35 U.S.C. § 103 as obvious over Smith.

ISSUE

The Examiner contends that Smith describes a method for cleaning one or more membranes normally immersed in water containing solids where one or more cleaning events occur per week over and at least 15 days that meet step (a) and step (b) of the claimed invention. The Examiner contends that the frequency of the cleaning event can be optimized depending on the quality and quantity of water to be treated (Answer 4).

The question before us is whether the Examiner has properly determined that the Smith reference teaches or describes the claimed subject matter within the meaning of 35 U.S.C. § 102(b) when the Examiner recognizes that the optimization of the cleaning process described in Smith is required to arrive at the claimed cleaning frequency feature. We answer this question in the negative.

PRINCIPLES OF LAW

The Examiner has the burden of making out a prima facie case of anticipation by pointing out where each and every element of the claimed invention is described in a single prior art reference, either expressly or under the principles of inherency, in a manner sufficient to have placed a

person of ordinary skill in the art in possession thereof. *In re Spada*, 911 F.2d 705, 708, 15 USPQ2d 1655, 1657 (Fed. Cir. 1990).

In rejecting claims under 35 U.S.C. § 103, the Examiner bears the initial burden of presenting a prima facie case of obviousness. *In re Rijckaert*, 9 F.3d 1531, 1532, 28 USPQ2d 1955, 1956 (Fed. Cir. 1993).

ANALYSIS

The Examiner has not carried the burden of making out a prima facie case of anticipation in the first instance is established by pointing out where each and every element of the claimed invention is described identically in the Smith reference, either expressly or under the principles of inherency. *See In re Spada,* 911 F.2d 705, 708, 15 USPQ2d 1655, 1657 (Fed. Cir. 1990). We agree with Appellants' description of the Smith reference appearing on page 5 of the Brief. Specifically, Smith Figure 4 describes three relevant chemical containing cleaning events (1, 5, and 6). Each cleaning event is an isolated event performed as part of the test to verify the effectiveness of the biocidal solutions (col. 18, ll. 30-40). Consequently, we agree with Appellants that even if cleanings (1) and (6) as specified in Figure 4 were the same as step (a) of the present invention, there is no disclosure of the subsequent step of performing one or more cleaning events as required by the claimed invention (Br. 5). This is especially true since the Examiner's discussion of optimization of the cleaning frequency depending upon the quality and quantity of water to be treated is an admission that the Smith reference does not describe each and every claim limitation.

As to the Examiner's rejection of claims 12-16, 36, 41, and 42 under 35 U.S.C. § 103, we note that the Examiner has not provided an analysis of

the limitations recited in independent claims 1 and 43. Since claims 12-16, 36, 41, and 42 encompass the limitations recited in the independent claims, we determine that the Examiner's analysis lacking any discussion of such limitations is incomplete. As such, we remand this application to the Examiner to provide a complete analysis of the Examiner's rejection of claims 12-16, 36, 41, and 42 under 35 U.S.C. § 103 as obvious over Smith and to reconsider the patentability of the remaining claims under 35 U.S.C. § 103.

CONCLUSION OF LAW

The Examiner erred in rejecting claims 1, 3, 35, and 43 under 35 U.S.C. § 102(b) as anticipated by Smith.

The application is remanded to the Examiner to provide a complete analysis of the Examiner's rejection of claims 12-16, 36, 41, and 42 under 35 U.S.C. § 103 as obvious over Smith and to reconsider the patentability of the remaining claims under 35 U.S.C. § 103.

ORDER

The rejection under 35 U.S.C. §102 is REVERSED and the application is remanded to the Examiner for appropriate action not inconsistent with the above instruction.

This remand to the examiner pursuant to 37 C.F.R. § 41.50(a)(1) (2004)) is not made for further consideration of a rejection. Accordingly,

37 CFR § 41.50(a)(2) does not apply..

REVERSED/REMANDED

sld/clj

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